nalytical bstracts

A monthly publication dealing with all branches of analytical chemistry: issued by the Society for Analytical Chemistry

ıme 6
12, Abstracts 4655–5073

ember, 1959

Research Library
JAN 11 1960
PEPSI-COLA COMPANY

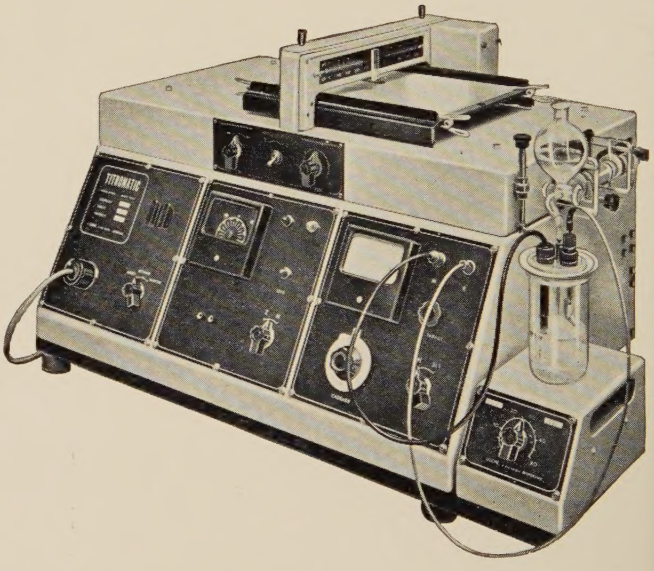
PLEASE RETURN TO
RESEARCH LIBRARY
PEPSI-COLA COMPANY

ed for the Society by
FFER & SONS LTD., CAMBRIDGE, ENGLAND

THE QUÉRE TITROMETER

has wide applications in recording physico-chemical processes

The Quére Titrometer is a precision instrument for recording titrations accurately. Equipped with an automatically variable recording table to suit the gradient of the curve, it permits the rate of advance to be reduced when the rate of change increases. Reagent flow is controlled by the table mechanism and by changing the jet of the syringe, the volume being represented by the abscissa of the graph and the state of mixture by the ordinate. The circle method determines accurately the end points of titration. *The circuits permit all kinds of potentiometric titrations and with special attachments it is possible to perform conductimetric or polarised-electrode titrations.*



✳ **PHOTOMETRY** The Quére strip photometer attachment is provided with filters and a vacuum type photocell giving accurate and stable readings.

✳ **USING A LIQUID MEDIUM** photometric attachment to this Recorder allows any photocolourimetric titration, recording photometric variations instead of equivalent points.

✳ **INTEGRATION** The Recorder automatically integrates the curve as it is traced. The results are entered on the axis of the ordinate of the curve by means of an electric pen marking 'tens' and 'units'.

✳ **RECORDING TIME FUNCTIONS** The Titrometer is fitted with a zero drift correcting circuit using an automatic potentiometer. To make accurate measurements it is necessary only to check the zero periodically; the instrument itself will then introduce the necessary corrections.

Write for fully illustrated and descriptive leaflet

Tinsley

H. TINSLEY & CO. LTD.

Werndee Hall, South Norwood, London, S.E.25

Tel.: ADDISCOMBE 6046-8

JSP 5719

Routine Values

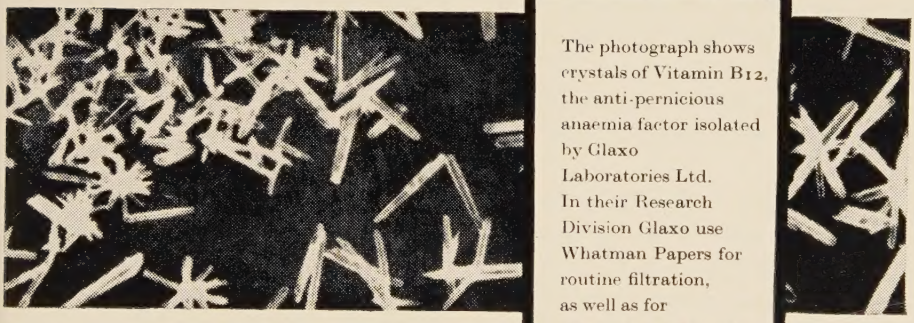
a concept of importance for all research workers

The concept signifies a painstaking empiricism, the thorough regard for routine detail, those less dramatic aspects of research. Concern for "routine values" covers many things, from the design of apparatus to the quality of research materials. It is as relevant in the development of papers for filtration as to work in any other branch of science or technology.

Applied for more than a century, the concept has set a seal of excellence on every Whatman Filter Paper.

The result is that *Whatman* has come to stand for the most reliable range of filter papers. They are an automatic choice of research organisations in Great Britain and in many other countries. They bring a consistently high standard of performance—a routine Whatman feature—to all filtration procedures.

The range of Whatman Filter Papers is discussed in the "Filtration Section" of the Whatman catalogue. Please write for a copy of this monograph and of the Whatman "Buyers' Guide."



The photograph shows crystals of Vitamin B_{12}, the anti-pernicious anaemia factor isolated by Glaxo Laboratories Ltd. In their Research Division Glaxo use Whatman Papers for routine filtration, as well as for chromatography and electrophoresis.

H. REEVE ANGEL & CO LTD

GT. BRITAIN 9 Bridewell Place London EC4

U.S.A. 9 Bridewell Place Clifton New Jersey

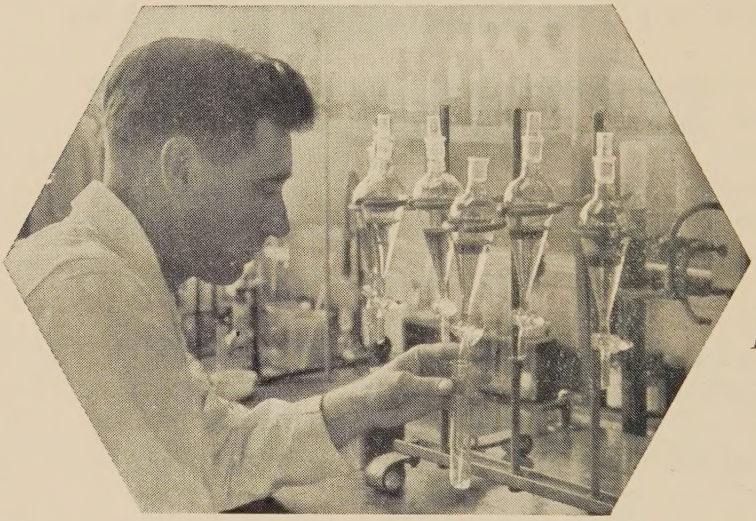
Sole distributors of

WHATMAN FILTER PAPERS

manufacturers W. & R. Balston Ltd.

Incidental information

No. 13



Items o
interes
from ou
laboratory
notebook

▶ Most analysts know about 1:10-phenanthroline and many use it for iron determinations. Not so many people seem to know that **4:7-diphenyl-1:10-phenanthroline** is twice as sensitive as 1:10-phenanthroline in the colorimetric determination of iron. There are several papers on the subject but the latest is *Analyst*, 1958, **83**, 80. The reagent is also called **Bathophenanthroline**, and we make it.

▶ Then, again the substitution of methyl groups in the 2:9 positions has the interesting effect of making the reagent insensitive to iron and we then have a selective and sensitive reagent for copper (see *Anal. Chem.*, 1956, **28**, 1158). Hopkin & Williams make **2:9-dimethyl-1:10-phenanthroline** (sometimes called **Neo cuproin**).

▶ One does not think of sulphate as radical one can determine absorptiometrically but this is now possible for low concentrations **Barium chloranilate** is the reagent and there ar two papers on the subject—*Anal. Chem.*, 1957 **29**, 281 and *Anal. Chem.*, 1958, **30**, 202 Hopkin & Williams make it.

▶ Hopkin & Williams Ltd. were also earl off the mark with supplies of the remarkabl new colour-producing reagent for fluoride ions **3-aminomethylalizarin-NN-diacetic acid**, des cribed by Belcher, Leonard and West (*Talanta* 1959, **2**, 92.) This important reagent is alread available from stock.

H&W

FINE CHEMICALS

for research and analysis

HOPKIN & WILLIAMS LTD., CHADWELL HEATH, ESSEX, ENGLAND

London, Manchester, Glasgow.

Agents throughout U.K. and all over the world

TAS/HW.

ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

eneral reviews of progress, reagents and methods of general application.

. Requirements for analysis by differential ion. H. H. Strain (Argonne National Lab., t, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 1.—The important features common to all ntial migration methods of analysis are re- and modifications and applications discussed. K. A. PROCTOR

. Diffusion analysis of ions in gels. M. nan, D. Eby and G. B. Kauffman (Hampshire cal Corp., Nashua, N.H., U.S.A.). *Anal.* 1959, **31** (5), 829–833.—Mixtures of common 1 0·1 *N* soln. are partly resolved by radial ntial diffusion in gels by means of chemical ial gradients. The gels are thick and made atin, poly(vinyl alcohol), or Na alginate. ethod is well suited to qual. analysis. For determinations place 0·05 ml (as a round on the central circle of the gel plate, measure dial distance traversed after known time- als, and compare with a calibration curve of ce *vs.* log concn. (Fick's law). For colourless se a developer. Thus $Pb(NO_3)_2$ (0·1 to 10 *N*) rmined (no details given) to within ± 0·7%, KI to arrest diffusion. For a series of halides own that the diffusion of the anion is affected e cation (Na, K or NH_4^+) present, though iffuse, and can be determined, independently. ction between ions and the gel may also interference. J. P. STERN

7. Use of amalgams in chemical analysis. ležal, B. Moldan and J. Zýka (Dept. Anal. , Charles' Univ., Prague). *Chem. Listy*, **53** (4), 398–408.—A detailed survey of the rties and use of amalgams in the reduction etermination of numerous elements is given. ferences.) J. ZÝKA

8. Schiff reagent of calibrated sensitivity. lftman (Columbia Univ., New York). *J. hem. Cytochem.*, 1959, **7**, 93–97.—The sensi- of Schiff's reagent for the detection of low of aldehyde increases as the concn. of H_2SO_3 reagent is decreased. The calibration of the ivity is readily accomplished by titration t iodine (Lugol's soln.). The stability of s soln. depends on decreasing the evaporation $_2$ and on retarding the change of sulphite to ate; to this end, refrigeration of a soln. ning more SO_2 than will be required, followed acuation of the excess of SO_2 or the use of cal preservatives (*e.g.*, 0·5% of quinol), are sed. Spectrophotometric measurements of loured soln. produced by adding aldehyde to s reagent indicates that at least two com- s may result; one has a violet colour but, when ncn. of aldehyde is so low that only a Schiff's t of low sulphurous titre will react with it, a red colour is produced; spectrophotometric analysis shows this to be different from the original basic fuchsine and validates its use as an end-point indicator in the reaction of aldehyde with Schiff's reagent. K. R. C.

4659. **Thiomalic [mercaptosuccinic] acid in inorganic analysis.** R. N. Sen Sarma (Jadavpur Univ., Calcutta). *Sci. & Cult.*, 1958, **23** (8), 434–435.—Preliminary investigations indicate that mercaptosuccinic acid (**I**) may be used as a standard reducing agent in volumetric analysis, and Fe^{3+}, Cu^{2+}, Mn^{7+}, Cr^{6+} and V^{5+} have been quant. determined by direct titration with an aq. soln. of **I** in weakly acid medium. **I** may be also be used as a colour-forming reagent, stable coloured products being formed with Fe^{2+}, Ni^{2+}, Co^{2+}, U^{6+} and Mo^{6+}. Iron, Mn, Pb, Bi, Hg and UO_2^{2+} are not pptd. with aq. NH_3 in the presence of an excess of **I**, which enables the rare-earth elements to be separated from them. Determination of Cu^{2+}, Fe, Mn^{2+} and Ni^{2+} can be made by a complexometric titration of the coloured products formed with **I**; the coloured system with Fe may be used as an indicator in the titration of Ca, Mg and Pb with EDTA. The use of **I** as a pptg. agent is discussed: Hg^{I} forms a black ppt., and Pb^{II}, Hg^{II}, Zn, Cd, Sn^{II}, Ag and W^{VI} form white ppt. in weakly acid soln. K. R. C.

4660. **Polarographic examination of the chelating power of EDTA and some closely related chelating agents.** W. Hoyle and T. S. West (Chem. Dept., Univ., Birmingham, England). *Talanta*, 1959, **2** (2), 158–164.—A rapid polarographic method is described for studying the stability of new complexones, in which a standard curve relates the half-wave potentials to the stability constants of a group of metal chelates. The selection of a suitable cation is discussed, and it is shown that only Cu^{II} and Cd, among common cations, are suitable, Cu being more satisfactory than Cd. From a study of the polarograms of a number of cupric chelates recorded at pH 2·5 and room temp., the chelate of di-(2-hydroxyethyl)glycine (DHEG), with $E_{\frac{1}{2}} = -0{\cdot}02$ and $\log_{10}K = 8{\cdot}1$, is chosen as zero reference standard, and a curve of $\Delta \log_{10}K$ *vs.* $\Delta E_{\frac{1}{2}}$ (where Δ is the difference between the chelate and DHEG values) is constructed, stability constants being taken from the literature. Values of $E_{\frac{1}{2}}$ are tabulated for 18 chelating agents, and $\log_{10}K$ values for 6 of these obtained from the curve. The curve appears to be linear for the known complexones, but this may not hold for newer reagents which are much more powerful in cation-binding power. The polarographic technique is described, and it is claimed that data on the chelating power of new reagents can be obtained in a few hours by the present method, compared with days by conventional methods. R. E. ESSERY

4661. **Complexones. XXX. Diethylenetriamine-penta-acetic acid (DTPA).** G. Anderegg, P. Nägeli and G. Schwarzenbach (Lab. f. anorg. Chem., Eidg. Tech. Hochsch., Zurich, Switzerland). *Helv.*

Chim. Acta, 1959, **42** (3), 827–836 (in German).—DTPA with metal cations M^{n+} forms complexes of the type MZ^{n-5}, MHZ^{n-4} and M_2Z^{2n-5} where Z^{5-} stands for the anion of DTPA. Bimetallic complexes of type $MM'Z^{n_1+n_2-5}$ are also formed. The stability constants of these species with Ca, Mn, Fe^{II}, Fe^{III}, Co, Ni, Cu, Zn, Cd, Pb, Hg and Ce were determined by using 7 methods. The DTPA complexes are considerably more stable than the corresponding EDTA complexes, making DTPA a better titrant in complexometry. G. P. Cook

4662. **Analytical applications of the complexones. XV. 1:2-Diamino*cyclo*hexanetetra-acetic acid as an analytical reagent.** F. Bermejo Martínez and R. Rey Mendoza (Lab. de Quím. Anal. de la Fac. de Ciencias, Santiago de Compostela, Spain). *Inf. Quím. Anal.*, 1959, **13** (2), 31–37.—The apparent oxidation potential of 1:2-diamino*cyclo*hexanetetra-acetic acid is a linear function of the pH of the soln. The reagent is not attacked by $K_2Cr_2O_7$, $KBrO_3$, iodine or H_2O_2 in acid soln. at room temp., but $KMnO_4$ and $Ce(SO_4)_2$ react under these conditions, as does iodine in alkaline soln. G. H. Foxley

4663. **Principles of end-point detection in chelometric titrations using metallochromic indicators. Characterisation of end-point sharpness.** C. N. Reilley and R. W. Schmid (Univ. of N. Carolina, Chapel Hill, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 887–897.—A numerical evaluation of end-point sharpness for chelometric titrations based on pM *vs.* pH and pY *vs.* pH diagrams is proposed. The use of these diagrams to predict optimum titration conditions and conditions suitable for selective titrations is discussed. The pertinent equilibria controlling end-point sharpness are enumerated, and their effects on pM *vs.* pH and pY *vs.* pH curves are illustrated by reference to the titration of Mg, Ba, Pb and Zn with EDTA and various indicators. J. P. Stern

4664. **Investigations on spot-test papers.** G. Ackermann (Inst. f. anorg. Chem. der Bergakad. Freiberg/Sachsen, Germany). *Mikrochim. Acta*, 1959, (3), 357–369.—Studies on 69 varieties of filter-paper showed that the sensitivity of a reaction can be influenced by the exchange capacity as well as by the surface area of the drop. The sensitivity of the detection of cations is mainly determined by the exchange capacity, whereas the surface area of the drop is the primary determining factor in anion detection. D. F. Phillips

4665. **Application of separating columns in analytical chemistry.** H. W. Kohlschütter, H. Getrost, G. Hofmann and H. H. Stamm (Eduard Zintl Inst. f. anorg. u. phys. Chem., Tech. Hochsch., Darmstadt). *Z. anal. Chem.*, 1959, **166** (4), 262–273.—Two substances in soln. may be separated by passage through a column if one of them undergoes some form of interaction with the column material. This has been applied to a study of the separation of Cl^-, SO_4^{2-} and PO_4^{3-} on a column of $Fe(OH)_3$, and to the separation of Zn^{2+} and Al^{3+} on a column of silica gel. T. R. Andrew

4666. **New redox volumetric agents. VII. Titrations with vanadium(II) sulphate.** J. Vulterin (Dept. Chem. Ind., Sch. of Chem. Engng, Prague). *Chem. Listy*, 1959, **53** (4), 383–391.—A review of methods for the volumetric determination of organic and inorganic compounds by me VSO_4 soln. is presented. (22 references.)

VIII. Titrations with vanadyl sulpha acetate. J. Vulterin. *Ibid.*, 1959, **53** (4) 397.—A survey of titrations with vanadyl acid and alkaline media is given. (25 refere J. Z

4667. **Use of potassium manganate in qu tive analysis. I. General.** G. den Boef (Anal Lab., Univ. Amsterdam, Holland). *Z. anal.* 1959, **166** (5), 321–325.—Oxidimetric titrat alkaline soln. (0·5 to 4 *M*) can be carried o manganate soln. Errors due to oxygen lo the reduction of $KMnO_4$ to MnO_2 by son stances are minimised by this method. B. B. Baumi

4668. **Coulometric analysis.** V. A. Mirkin. *Lab.*, 1959, **25** (3), 292–296.—A review is with 67 references. G. S. S

See also Abstract—**5042**, Analytical poss of micro-wave spectroscopy.

2.—INORGANIC ANALYSIS

General, determination of elements (arrang in the order of the Periodic Table), analysis minerals and inorganic industrial produc

4669. **Separation and micro-identification o ions by solvent-extraction and ring-oven tech** P. W. West and Anil K. Mukherji (Louisian Univ., Baton Rouge, U.S.A.). *Anal. Chem.* **31** (5), 947–950.—The combination of s extraction and ring-oven techniques perm separation and identification of 35 cations drop of aq. soln. The aq. cation chlorides HCl are extracted with *iso*butyl methyl k amyl acetate (2:1), when Sb, Au, Fe, V, M As, Ge and Te appear in the ring formed organic phase. The aq. phase is then trea pH 2 with 7 *M* NH_4SCN (3 drops) and ex with diethyl ether; the ether contains Co, and Sn. The aq. phase is then slowly ex with acetylacetone, first at pH 2 (to extra Cr, Cu, U, Zr and Ti) and then at pH 7 (to co the extraction of Al). The aq. phase i treated at pH 3 with 2% Na diethyldithiocarl soln. and immediately extracted with diethyl Ni, Cd, Bi, Pb, Hg, Tl, W, Mn, Se and In pa the ether. The final aq. residue contains Ca, Mg, Ce and Th. Spot tests for the identif of the individual cations in the rings are det J. P. S

4670. **Ion-exchange separations. Chr graphic separations based on ion charge.** Fritz and S. K. Karraker (Iowa State College, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 921- The greater affinity of a cation-exchange re a tervalent than for a bivalent cation perm quant. separation of Zn from La. Thus 0·2 mole of Zn is separated from 2·5 millimoles with a recovery > 99·4%. Bivalent ionic (*e.g.*, ethylenediammonium chloride soln.) used in lower concn. as eluents than un liquids (*e.g.*, HCl). *Procedure*—Add the Zn soln. (approx. 0·01 *M* at pH < 3) (25 ml) at per min. to the resin column (Dowex 50-X8 diam. 12 or 21 mm) previously treated with

ıediammonium chloride. Elute with 0·1 *M* ıediammonium chloride (pH 3) and collect fractions. Determine Zn and La by titration 05 *M* EDTA, with "naphthylazoxine" [7-(1-ıylazo)-8-hydroxyquinoline-5-sulphonic acid 'senazo indicators, respectively.
J. P. STERN

. **Paper chromatography in the separation of III. Separation of silver, copper and arsenic elements.** A. K. Majumdar and M. M. ıbartty (Jadavpur Univ., Calcutta). *Anal. Acta*, 1958, **19** (2), 132–134.—The constants e metal ions Hg^{2+}, Ag^{+}, Pb^{2+}, Tl^{+}, Cu^{2+}, Bi^{3+}, As^{3+} and Sb^{3+} are determined by the que and with the solvents previously used *Abstr.*, 1959, **6**, 2177). The separation of res containing up to five components appears e but, with the exception of NH_4I, the com- g agents give little advantage H. N. S.

2. **Determination of aluminium, titanium ckel in their alloys.** L. E. Hibbs and D. H. ıs (Res. Lab., General Electric Co., ectady, N.Y., U.S.A.). *Talanta*, 1959, **2** (1), —The alloy is dissolved in aqua regia and, lestroying HNO_3, the soln. is passed through mn of Dowex-1 anion-exchange resin; Al, Ni i are then eluted with 7 *M* HCl, leaving Co, l Fe on the resin. The eluate containing Al, d Ti is then passed through two columns cted in series containing, respectively, a ic resin and an anionic resin. On eluting ·8 *M* HF - 0·06 *M* HCl the Al passes through olumns, the Ni is retained on the cationic resin ıe Ti on the anionic resin. The columns are nected and Ni is eluted with 4 *M* HCl and h 3 *M* HCl. The metal content of each of ree fractions is determined by titrating with and standard Cu soln. at pH 4·8, with ı as fluorescent indicator (*cf.* Wilkins, *Anal.* 1959, **6**, 4419). W. T. CARTER

3. **Amperometric titration method for the is of mixtures of bismuth, aluminium, calcium magnesium salts.** Yuen-Kai Oong and Chen Lee (Dept. of Anal. Chem., Nanking ı. Coll.). *Acta Pharm. Sinica*, 1959, **7** (3), 3.—The amperometric titration with 0·1 *M* at controlled pH and potentials (*cf.* Reilley *et nal. Abstr.*, 1956, **3**, 2401) has been applied to res of the salts of Bi, Al, Ca and Mg. For the test soln. is adjusted to pH 1·5 to 2 with NaOH and solid chloroacetic acid. The on with EDTA is carried out in 0·0025% ı soln. and in the absence of air, at the applied ıial of − 0·25 V *vs.* the S.C.E. For combined Ca^{2+} and Mg^{2+}, after titration of Bi^{3+}, the s treated with an excess of EDTA, adjusted 4 and heated almost to boiling. The soln. ed and adjusted to pH 8 with aq. NH_3. The of EDTA is then back-titrated with 0·05 *N* $)_3)_2$ at the applied potential of + 0·05 V *vs.* .C.E. For combined Ca^{2+} and Mg^{2+}, tri- ɔlamine is added to the test soln. to mask l Bi, and the pH is adjusted to 10 with 6 *N* before titration with EDTA at the applied ıial of + 0·02 V *vs.* the S.C.E. For Ca^{2+}, ıre previously removed from the test soln. H 4) as sulphide. The soln. is then adjusted 8 with dilute KOH soln.; Zn^{2+} are added to as indicator. The titration with EDTA is l out in the absence of air at the applied ıial of − 1·6 V *vs.* the S.C.E. S. H. YUEN

4674. **Determination of nickel, zinc and aluminium in bronze and brass after determining copper, lead, tin and antimony by controlled-potential electrolysis.** B. Alfonsi (Lab. Auto-Avio, FIAT, Turin, Italy). *Anal. Chim. Acta*, 1959, **20** (3), 277–282.—Procedures are given for the successive determination of Ni, Zn and Al in the acid soln. remaining after the electrolytic separation of Cu, Pb, Sn and Sb (*Ibid.*, 1958, **19**, 276, 389, 569). The Ni is pptd. with dimethylglyoxime in slightly ammoniacal soln. at 45° to 50°, and the Zn in the filtrate is separated by controlled-potential electrolysis (0·4 to 0·8 amp., − 1·2 to − 1·55 V *vs.* the S.C.E.) in the presence of hydrazine hydrochloride. The Al is then determined in the final soln. by the addition of 8-hydroxyquinoline, dissolution of the oxinate in conc. H_2SO_4 and, after addition of $HClO_4$ and removal of any SiO_2, re-pptn. with ammonium benzoate at pH 3·6 to 4·2 in the presence of thioglycollic acid. Zinc should not be determined electrolytically if $>$ 10 mg of Fe is present; if there is $<$ 10 mg of Fe the amount co-deposited with the Zn should be determined. The accuracy is adequate for routine analysis. W. J. BAKER

4675. **Analysis of fission-product mixtures.** E. L. Geiger (Reynolds Electrical and Engng Co., Las Vegas, Nev., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 806–809.—A procedure is described for the rapid, sequential separation of radioactive Ru, Sr, rare-earth metals, Zr - Nb and Cs in air-filter, soil, sediment, vegetable and aqueous samples. The fission products are leached from the samples with a 3 *N* HCl - 0·1 *N* HF soln. and recoveries of 97% and individual decontamination factors $> 10^3$ are obtained. A complete analysis requires 16 hr., and the precision of each analysis is within $\pm$ 4% at the 90% confidence limit. K. A. PROCTOR

4676. **Photometric determination of traces of hydrogen peroxide and copper(II) ions with phlorrhizin [phloridzin].** L. Erdey and J. Inczédy (Tech. Univ., Budapest). *Mikrochim. Acta*, 1959, (3), 438–444 (in German).—The increased oxidation potential of H_2O_2 in the presence of certain metal ions forms the basis of a sensitive colour reaction of phlorrhizin in the presence of Cu^{2+}, which is suitable for the photometric determination of H_2O_2 at pH 12. Similarly, traces of Cu^{2+} can be determined at pH 9.
D. F. PHILLIPS

4677. **Relation between radiation intensity and concentration of the alkali metals in flame photometry.** N. S. Poluéktov and M. P. Nikonova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (3), 263–268.—Apparent discrepancies between the experimental and theoretical shapes of the log intensity *vs.* log concn. curves are explained theoretically. G. S. SMITH

4678. **Influence of certain elements on the determination of the alkali metals by flame photometry.** T. K. Aĭdarov, A. E. Zak and E. S. Safonova. *Zavod. Lab.*, 1959, **25** (3), 269–271.—The effects of Na, K and Ni on the determination of Li, and the effects of Cd and Ni on the determination of K are studied, with particular reference to the analysis of electroplating soln. G. S. SMITH

4679. **Flame-photometric determination of oxides of alkali metals in tungstic acid.** R. Hamerník (Res. Lab., Tesla, Rožnov, Czechoslovakia). *Hutn. Listy*, 1959, **14** (2), 142–144.—Potassium and sodium can be separated from tungstic acid by heating the

sample with water acidified with HCl. The presence of sodium increases the colour intensity of the potassium flame, but a similar effect of potassium on sodium was not observed. Small amounts of Ca do not interfere. *Procedure*—Transfer the sample ($\simeq$ 2 g) to a 250-ml flask of resistance glass or silica, and add H_2O (30 ml) and conc. HCl (5 to 7 drops). Heat under reflux for 2 hr. Separate the tungstic acid by filtering, wash with a small vol. of slightly acidified H_2O (5 drops of conc. HCl in 100 ml of H_2O), dilute the filtrate to 100 ml and measure with a flame photometer. The error is $\not>$ 0·002% of K_2O or Na_2O. J. ZÝKA

4680. **Determination of lithium and potassium chlorides in the presence of each other by electrical conductivity.** J. Zatloukal (Res. Inst. Inorg. Chem., Ústí nad Labem, Czechoslovakia). *Chem. Průmysl*, 1959, **9** (3), 131–132.—The total amount of both elements is determined by evaporation. The residue is dissolved in a known vol. of H_2O, and the electrical conductivity is determined and compared with a calibration curve of standards containing various ratios of LiCl and KCl at constant total concn. For a ratio of LiCl to KCl of 1:1 an accuracy within $\pm$0·2% has been achieved, which changes to $\pm$1% if LiCl to KCl is 1:10, and to $\pm$10% if LiCl to KCl is 1:100. A Wheatstone bridge supplied with a.c. at 4 V and 50 c/s is used for the measurement and a Tesla Service oscillograph (with a transformer for increasing the sensitivity) for the indication of the balance point. J. ZÝKA

4681. **Flame-photometric determination of small amounts of sodium in silumins.** J. Weiss and B. Bieber (Res. Inst. of Materials and Technol., Brno, Czechoslovakia). *Hutn. Listy*, 1959, **14** (3), 247–248.—*Procedure*—Dissolve the finely ground sample (0·5 to 2·0 g) by boiling in a small porcelain beaker in HCl (1:1) (20 ml), cool, transfer to a 100-ml flask, dilute to vol. with twice-distilled H_2O, set aside and in an aliquot of the supernatant liquid (graphitic Si remains in the ppt.) determine the Na content with a Zeiss Model III flame photometer (filter Na 59, shade 40). Refer the results to a calibration curve. The procedure can be carried out in $<$20 to 30 min. with an accuracy of 0·002% of Na in samples containing 0·01 to 0·001% of Na. The results are not influenced by the presence of Mn (0·70%), Fe (1·50%), Ti (0·15%), Cu (1·80%), Zn (1%) or Mg (0·50%). For the determination of Na in the presence of Al, calibration curves for 0·5, 1·0 and 2·0 g of the sample with added $AlCl_3$ must be prepared. A suitable method is proposed for the preparation of Na-free $AlCl_3$ by crystallisation and pptn. in a current of HCl. J. ZÝKA

4682. **Adaptation of Winkler's method to the routine determination of alkali carbonate and hydroxide.** Z. Hošťálek (Dept. Inorg. Technol., Sch. of Chem. Technol., Prague). *Chem. Průmysl*, 1959, **9** (3), 123–125.—The sources of error in Winkler's method (the influence of phenolphthalein, pptn. of $BaCO_3$, addition of $BaCl_2$, influence of CO_2, decomposition of $BaCl_2$ with acid, concn. of acid and vol. of the soln.) have been systematically studied. The errors are influenced mainly by the ratio of the components to be determined. Conditions have been found for obtaining reproducible results and an empirical correction graph is given which eliminates systematic errors for the range of 0 to 95% of CO_3^{2-} in the total anions. For samples containing $>$95% of CO_3^{2-}, Warder's meth preferred. *Procedure*—Dilute 25 ml of the s soln. to 75 ml with H_2O, add *N* $BaCl_2$ (neutralised with $Ba(OH)_2$ or HCl to phenolphth then phenolphthalein soln (1% in 96% et (0·3 ml), mix thoroughly and titrate immed with 0·5 *N* to *N* acid added dropwise. The occupied must not exceed 3 min. Add m orange soln. (0·1%) (2 to 3 drops) and titrate stirring till the soln. is red (pH about 3·9). the results to a graph of observed values fo expression $(CO_3^{2-}/CO_3^{2-} + OH^-) \times 100$ *vs.* th values. J. Z

4683. **Solubility of potassium tetraphenyl in acetone - water solutions.** A. D. Scott, H Hunziker and M. G. Reed (Agronomy Dept., State Coll., Ames, U.S.A.). *Chemist Analyst*, **48** (1), 11–12.—Results for the solubility tetraphenylboron (**I**) at 28° for acetone - mixtures from 16 to 100% (v/v) of aceton tabulated and presented as a graph. The solu rises sharply from 1·2 mg per ml in a 32% so 59·8 mg per ml in a 95% soln., and falls ra thereafter to 41·8 mg in 100% acetone. analytical procedures in which pptd. **I** is diss in acetone and titrated with aq. soln., the ace water ratio should be kept as high as the ditions allow. R. E. Ess

4684. **Photometric determination of copper diaminocyclohexanetetra-acetic acid [Compl IV].** W. Nielsch (Karl-Reiss-Platz 12, Mann Germany). *Mikrochim. Acta*, 1959, (3), 419–4 In the pH range 5 to 11, Complexone IV forms Cu^{2+} a complex that has a max. extincti 720 mμ, which is independent of the type of used and also of the acid used for dissolvin metal (*e.g.*, sheet copper). A favourable slo the calibration curve is obtained. The meth suitable for the determination of Cu in the c range 20 to 110 μg per ml. No precision is q D. F. PHILI

4685. **Trace determination of copper and by chelate chromatography and dithizone p metry.** D. M. Smith and J. R. Hayes (Pe vania State Univ., University Park, U. *Anal. Chem.*, 1959, **31** (5), 898–902.—Coppe Co ($\simeq$ 10 μg) are separated from each othe from Fe^{3+} (10 mg), Mn (5 mg), Zn (10 mg (0·1 mg) and Ni (50 μg) by chromatography diethyldithiocarbamates on silica gel, with C CCl_4 (1:1) (**I**) as developer. The element determined photometrically as dithizones i eluate. *Procedure*—To the aq. metal nitrate aq. NH_3 - NH_4Cl buffer (pH 9·6 to 9·8) (2 10% $Na_4P_2O_7$ soln. (15 ml) and 0·1% Na di dithiocarbamate soln. (2 ml), and after 1 extract with CCl_4 (3 × 3 ml). Pass the ex through 3 g of silicic acid activated at 500° hr.; the flow rate should be 0·5 ml per min. th a column 7·5 cm deep. Elute the Cu and Co **I** for 1 to 2 hr. (flow rate 0·3 to 0·4 ml per Destroy the complexes with conc. H_2SO_4 (0· and 60% $HClO_4$ (0·3 ml) at 200° to 250°. I and neutralise the soln. and convert the Cu Co into the dithizones for photometry at 51 545 mμ, respectively. J. P. ST

4686. **Concentration of traces of silver con in lead by a precipitation method.** Z. Za and O. Kempiński (Anal. Lab., Central La Accumulators and Cells, Poznan, Poland).

, *Warsaw*, 1959, **4** (1–2), 422–432.—Errors o the adsorption of AgCl on the $PbSO_4$ ppt. minimised by the use of *p*-dimethylamino-ylidenerhodanine for the concentration of Ag ptn. This method, in conjunction with a ographic determination of the Ag, provides a l means of determining the amount of Ag 0005%) in all commercial grades of lead, and oys containing Sb and in metallic cadmium. s are > 4% and the sensitivity of the method be increased about 50 times by increasing the le and diminishing the volume of polaro-ic soln. to the micro-analytical scale. The od is useful in controlling the purification of salts, and in the preparation of spectral lards. W. B. Miaskowski

87. **Determination of copper, iron and lead etallic silver.** W. Kemula, K. Brajter, S. k and H. Lipińska (Dept. of Inorg. Chem., ., Warsaw). *Chem. Anal., Warsaw*, 1959, **4** , 409–415.—By the method described, 0·02 to % of Cu, 0·006 to 0·01% of Fe and ≃0·003% o can be determined. The sample is dissolved nc. HNO_3, evaporated and diluted with H_2O. soln. is passed through a cation-exchange nn (Wofatit KPS-200), which is then treated ssively with *N* KCl to precipitate AgCl, ammonium salicylate to elute Fe^{3+} and with to elute Pb^{2+} and Cu^{2+}. The latter eluate is d to an anion-exchange column (Wofatit L); Pb is retained and can be eluted with *N* HNO_3. The separated cations are deter-d polarographically—Fe^{III} in *N* aq. NH_3 and onium salicylate as supporting electrolyte, Cu 5 *N* aq. NH_3 with 1·5 *N* NH_4Cl, and Pb in *N* O_3. W. B. Miaskowski

88. **Detection of beryllium with acetylacetone.** D. Cool (Madison Coll., Harrisonburg, Va., A.). *Chemist Analyst*, 1959, **48** (1), 7–8.— e a drop of test soln. on a slide and invert it conc. aq. NH_3. Remove the liquid from the . $Be(OH)_2$ with a capillary, and treat the ed residue with just enough satd. aq. acetyl-one soln. to dissolve the ppt. When oily drops ar, or crystallisation starts at the edge of the , draw a fine-tipped glass rod through the oily rystallised area and across the drop. In the ence of Be, characteristic right-angled crystals ar, which are examined under a magnification 50, along the track of the rod. The limit of in detection is 0·1 μg. When 0·5- and 1-μg nts of Be were used with 10 times these nts of other metals, interference was observed Al, Bi, As^V, Ce, Cr, Ga, In, Fe, La, Mn, Nd, Ru, Sm, Sc, Tm, Sn^{II}, Sn^{IV} and Zr. No inter-ce was observed with As^{III} and 43 other ls. R. E. Essery

89. **Quantitative spectrographic analysis of nesium alloys.** J. Fijalkowski (Lab. of Aviation erials, Aviation Inst., Warsaw). *Chem. Anal., saw*, 1959, **4** (1–2), 455–461.—A modified hod is described permitting the simultaneous rmination of alloy components, *e.g.*, Al, Zn Mn, and impurities, *e.g.*, Fe, Cu, Si and Ni. ils are given of the equipment used, the con-ns of analysis, and the results obtained by ng various line-pairs. The coeff. of variation ndividual elements ranges from ±4 to 7%, pt for Si, when it reaches 9%. W. B. Miaskowski

4690. **Electrochromatography in the separation of ions. VII. Separation of alkaline-earth metals.** A. K. Majumdar and B. R. Singh (Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1959, **20** (3), 275–277.—By using the procedure described previously (Majumdar and Mukherjee, *Anal. Chim. Acta*, 1956, **15**, 547), complete separation of ternary mixtures of Ca^{2+}, Mg^{2+} and Sr^{2+} or Ba^{2+} can be made with 0·1 *N* NH_4Cl as electrolyte, and less complete but detectable separation with 0·1 *N* NH_4NO_3. Binary separations (Ca - Ba, Ca - Sr, Ca - Mg, Mg - Sr and Mg - Ba) can be achieved with other electrolytes, *e.g.*, HNO_3, NaCl, acetic acid, aq. thiourea and Na rhodizonate. Movements (in cm) of the ions are listed. W. J. Baker

4691. **Visual EGTA [1:2-di-(2-aminoethoxy)-ethane-NNN′N′-tetra-acetic acid] titration of calcium in the presence of magnesium.** F. S. Sadek, R. W. Schmid and C. N. Reilley (Dept. of Chem., Univ. of N. Carolina, Chapel Hill, U.S.A.). *Talanta*, 1959, **2** (1), 38–51.—Calcium can be selectively titrated in the presence of Mg with EGTA, with zincon (2-carboxy-2′-hydroxy-5′-sulphoformazylbenzene) as indicator. The Mg can then be titrated in the same soln. with EDTA (disodium salt). *Procedure*—Neutralise the sample soln. and adjust to pH 9·5 with aq. NH_3 - NH_4NO_3 buffer. Add zincon soln. (0·2%) (2 to 3 drops) and sufficient of a soln. of EGTA (containing the stoicheiometric amount of Zn) to give a permanent blue colour. Titrate the Ca with a standard soln. of EGTA. Add KCN soln. and titrate the Mg with standard EDTA (disodium salt) soln., with Eriochrome black T as indicator. When Zn is present it can be determined by titration with standard tetraethylenepentamine dihydrosulphate soln. and zincon before the titration of Ca. The procedure can be used for the analysis of soln. containing Ca and Mg in ratios of up to 1:20, with an accuracy of ± 0·2% at the 5-mg level. A theoretical treatment is given of the equilibria involved in the titrations. W. T. Carter

4692. **New sensitive spot reaction for zinc.** V. Anger (Lab. Dr. V. Anger, Vienna 18). *Mikrochim. Acta*, 1959, (3), 473–475.—The detection of 0·05 μg of Zn in neutral or acid soln. at a limiting concn. of 1 p.p.m. is possible with trisodium pentacyano-NN-dimethyl-*p*-nitrosoanilinoferrate. In the presence of Zn a green ring appears which, on spotting with 2 *N* H_2SO_4, changes to blue, while the pale-green colour of the reagent is completely discharged. Mercury, Cu, Cd, Co, Ni, Fe and UO_2^{2+} interfere through formation of dark-green rings; Fe^{3+} may be masked by fluoride. Zinc may be detected in the presence of Cu, Cd and Ni after treatment of the ammoniacal test-soln. with colourless ammonium sulphide and subsequent separation of the ZnS with 2 *N* H_2SO_4 or HCl. This enables 2 μg of Zn to be detected in the presence of 1500-fold amounts of Cu, Cd and Ni. The reagent may also be used to detect 0·5 μg of Cd in the presence of 100-fold amounts of Cu. D. F. Phillips

4693. **Use of EDTA to eliminate interference in the zinc - ferrocyanide titration.** D. G. Davis and H. T. McLendon (Sch. of Chem., Georgia Inst. of Technol., Atlanta, U.S.A.). *Talanta*, 1959, **2** (2), 124–127.—The use of EDTA as a complexing agent for interfering metal ions is studied. For the detection of the end-point, the relative merits of potentiometric methods and of amperometric methods, using a manual polarograph and either one or two indicator electrodes, are discussed.

Amperometric methods are easier and more accurate. If the pH is controlled at 2·0 to 2·1, EDTA, sufficient to complex all the interfering metals, with an excess equivalent to approx. one-half of the Zn, eliminates interference due to Ni, Fe and Pb, while Al, Mn, Cl^- and SO_4^{2-} do not interfere either in the presence or absence of EDTA. Interference by Cu cannot be removed by EDTA. Thiosulphate is not satisfactory for masking Cu, while hydrazine sulphate is only useful when the Cu is much smaller in amount than the Zn. For most alloys, it is best to remove Cu by electrolysis, and then eliminate the remaining interferences with EDTA. R. E. Essery

4694. **Use of complexometric titration in the control of galvanising baths.** I. Dubský (Jablonecká Bižuterie n.p., Jablonec n. Nis., Czechoslovakia). *Chem. Průmysl*, 1959, **9** (2), 80–84.—Cyanide complexes of Zn and Cd react with acetone, ethyl methyl ketone, *cyclo*hexanone or benzaldehyde, and the liberated Zn^{2+} or Cd^{2+} can be titrated with EDTA (disodium salt) (**I**). The complexes of Cu, Ni, Co, Fe and Hg do not react in a similar way. *Procedure for Zn*—Dilute the sample to 10 to 30 ml, add pure acetone (30 to 50 ml), buffer soln. (pH 10) (NH_4Cl - aq. NH_3), and Eriochrome black T as indicator and titrate with **I** till blue. When titrating Cd an excess of **I** is added and the excess back-titrated with $MgSO_4$ soln. J. Zýka

4695. **Detection of cadmium by selective paper chromatography.** M. Ziegler and H. Winkler (Anorg.-chem. Inst., Univ., Göttingen). *Z. anal. Chem.*, 1959, **166** (4), 241–242.—Cadmium (5 μg) may be detected in the presence of Cu^{2+}, Pb^{2+}, Bi^{3+} (all in 2000-fold excess) or Hg^{2+} (100-fold excess) by transferring a drop of soln. to a paper strip and developing it for 1 hr. with a solvent prepared by shaking 3 vol. of *n*-butanol with 1 vol. of HCl (3 *N*) and saturating the organic phase with H_2S. Only Cd^{2+} migrate under these conditions, and the spot is detected by spraying the paper with $(NH_4)_2S$ soln. T. R. Andrew

4696. **High-frequency titrations. II. The mercury(II) - thiocyanate reaction.** Anil K. Mukherji and Bharat R. Sant (Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (3), 259–262.—With the apparatus and procedure described previously (Sant and Mukherji, *Ibid.*, 1959, **20**, 124), the oscillometric titration of $Hg(NO_3)_2$ soln. with aq. KSCN soln. gives one inflection corresponding to the quant. formation of $Hg(SCN)_2$, whilst in the reverse titration $Hg(SCN)^+$ are formed. This method can be used for the rapid micro-determination of either Hg^{2+} or SCN^- in very dil. soln. The accuracy is high; only in the range of 0·00003 to 0·00006 *M* does the error become 2 to 4%. The titration can be made in the presence of HNO_3 provided that the total acidity in the system is $\gtrsim$0·003 *N*. W. J. Baker

4697. **Direct thermometric titration of boric acid.** F. J. Miller and P. F. Thomason (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn., U.S.A.). *Talanta*, 1959, **2** (2), 109–114.—Apparatus and procedure are described for the automatic thermometric titration of H_3BO_3, a thermistor and bridge with automatic recording being used (*cf.* Linde *et al.*, *Brit. Abstr. C*, 1953, 330). The chart records the increase of potential as the titration proceeds, and the distance travelled by the chart is calibrated in terms of the vol. of titrant added from the burette. The beginning and end of the titr are marked by turning-points on the c Although not essential for a good titration addition of mannitol improves the shape o curve. In the presence of strong acid, both and the H_3BO_3 can be titrated sequent Amounts of H_3BO_3 in the region of 0·3 milli-e can be determined with a coeff. of variation of R. E. Esse

4698. **Determination of boric acid in m borate.** I. I. Strizhevskiĭ (All-Union Sci. Res. of Autogenous Treatment of Metals). *Z Lab.*, 1959, **25** (2), 146.—Free boric acid is d mined from the amount of water (3 moles mole of boric acid) obtained by reaction methanol in the presence of pyridinium iodide G. S. Smi

4699. **Separation of aluminium and iron copper, nickel, zinc and manganese on catio Application to the analysis of copper alloys.** Marczenko (Anal. Dept., Inst. of Gen. C Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (437–444.—By the use of a column of cati (Wofatit P, Dowex 50 or SBS, ammonium for is possible to separate, with a high degre accuracy, Al and Fe from Cu, Ni and Zn, u sulphosalicylic acid as complexing agent. results in the separation of Al plus Fe fron plus Ni plus Zn were obtained on all three catio in strongly ammoniacal medium, and on Wo P and Dowex 50 also in weakly ammon medium (pH $\simeq$ 9). In each case an attemp separate Al plus Fe from Mn proved unsucces W. B. Miaskows

4700. **Study of the chemistry of scandium tracer scale with scandium-46.** B. C. Purkaya and V. R. Pai Verneker (Inst. of Nuclear Phy Calcutta, India). *Anal. Chem.*, 1959, **31** (5), 815.—A study of the mixed crystal formatio ferric sulphate containing tracer amounts o Y and Eu indicates that Sc has a greater resembl to the iron family than to the rare-earth elem K. A. Proct

4701. **Spectrophotometric determination of ce in fluoride salts.** J. C. White and R. F. A (Anal. Chem. Div., Oak Ridge Nat. Lab., T U.S.A.). *Talanta*, 1959, **2** (2), 176–182.—Terv Ce in fluoride salt mixtures is determine measurement of its u.v. peak at 253 mμ in HCl. Weigh the sample (2 to 5 g) into a plati dish, add 1 g of H_3BO_3 and mix. Add conc. H (5 ml), conc. HCl (20 ml) and conc. H_2SO_4 (2 and heat to strong fumes. Cool, and dilut 500 ml with water. Transfer an aliquot ($\gtrsim$ 1 containing 0·5 to 5 mg of Ce) to a centrifuge Add conc. aq. NH_3 dropwise till the Ce is p and 2 ml in excess. Centrifuge for 2 min., de the supernatant liquid and dissolve the pp 10 ml of 7 *M* HCl, making up to 25 ml with same acid. Transfer to a separating-funnel, 5 ml of 0·05 *M* tri-*n*-octylphosphine oxide in hexane, shake for 2 min., and allow to sepa Measure the extinction of the aq. phase ag water at 253 mμ. Beer's law is obeyed up to 0· of Ce per ml. Interfering elements are rem either by the pptn. or the extraction, but Cr reduced to Cr^{III} in 7 *M* HCl, and this is not ex ted. Chromium in amount three times that o Ce introduces a 4% error, but it is rarely pr in fluoride salts in amounts large enough to

ty. Over a range from 0·4 to 8·9% of Ce, rerage difference between results by the : method and radio-tracer methods was
R. E. Essery

. **X-ray emission spectrographic analysis of urity rare-earth oxides.** F. W. Lytle and Heady (Rare and Precious Metals Expt. ı, U.S. Bureau of Mines, Reno, Nev.). *Anal.* 1959, **31** (5), 809–811.—Small amounts ɔ 1·0%) of rare-earth elements can be deter- by this method with an average error of ≃ The method is dependent on the accurate ination of the spectral line intensity above ckground. The preparation of the sample andards, the choice of analytical lines and e of a helium atmosphere for the optical ɔ increase the intensities are discussed.
K. A. Proctor

. **Emission spectrographic double-arc analy- readily volatile trace elements in graphite.** da and E. Schroll (Mineralog. Inst., Univ., ı). *Mikrochim. Acta*, 1959, (3), 389–401.— rocedure of Ahrens ("Quantitative Spectro- al Analysis of Silicates," 1954, London) for -arc analysis has been modified and applied determination of As, Ag, Sb, Cd, Cu, Ga, g, Mo, Pb, Sn, Tl and Zn present as traces phitic and carbonaceous rocks. A special was devised for the Ahrens-type micro te crucible and the work was carried out in s Qu 24 medium quartz spectrograph, with a step filter (100, 20 and 4% transmission). amples were prepared by mixing the finely l substance with Li_2CO_3 and $(NH_4)_2SO_4$ in oportions of 15:3:1, respectively, to give a crucible charge of 0·4 g. Excitation was by rc at 110 V and 7 to 10 amp. Detection of from 0·03 to 30 p.p.m. were attained. ing photometric evaluation with background tion, reproducibilities of ± 2 to ± 16% were ed for individual exposures.
D. F. Phillips

. **Rapid method for determination of small nts of carbon monoxide in gas mixtures.** syj, J. E. Zarembo and A. Hanley (Food nery and Chemical Corp., Princeton, N.J.,). *Anal. Chem.*, 1959, **31** (5), 902–904.— weighing of the CO_2, produced by the ion of CO over a decomposed $AgMnO_4$ st, and absorbed on Ascarite, permits 0·3 to of CO to be determined with an accuracy ± 0·85% in a gas sample (500 ml) containing to 78%), O (18 to 21%) and unsaturated carbons (1 to 5%). The absorption train ises Mersil (164 g of red HgO dissolved in l of cold 29% H_2SO_4; in this soln. is dissolved of $MgSO_4.7H_2O$, the product is filtered and with enough silica gel to give a moist mass, is dried at 100°) and anhydrone - Ascarite tion bulbs, a combustion tube containing O_4 at 450° to 500°, an anhydrone absorption the weighed Ascarite absorption tube, and a tube. The train is purged with O (20 to per min. for 10 to 15 min.) and the sample passed at 20 to 30 ml per min.; finally the s purged again with O for a further 15 min.
J. P. Stern

. **Rapid determination of carbonates.** F. L. (Angel Urraza 718–303, Mexico 12, D.F.,). *Z. anal. Chem.*, 1959, **166** (4), 243–246.— CO_3^{2-} content of washing powders, etc., has been determined by evolution of CO_2 with acid and subsequent absorption in NaOH so ln. in a simple apparatus.
T. R. Andrew

4706. **Determination of silicic acid by distillation of fluorosilicic acid in a silver apparatus.** P. Ehrlich and T. Keil (Anorg. Abt. des Chem. Inst. der Univ. Giessen). *Z. anal. Chem.*, 1959, **166** (4), 254–261.—From 10 to 150 μg of SiO_2 may be determined in the presence of 1 g of TiO_2, V_2O_5, ZrO_2 or Al_2O_3 by the steam-distillation of H_2SiF_6 from a soln. containing H_2SO_4 and HF in a silver apparatus, and subsequent photometric determination as molybdenum blue. The distillate is neutralised to *p*-nitrophenol and to an aliquot is added 15 ml of H_3BO_3 - $(NH_4)_6Mo_7O_{24}$ soln. [25 g of H_3BO_3 30 ml of HCl (sp. gr. 1·16) and in 400 ml of H_2O mixed with 10 g of $(NH_4)_6Mo_7O_{24}.4H_2O$ in 250 ml of H_2O and diluted to 1 litre] and the mixture is set aside for 5 min.; 10% citric acid soln. (4 ml) is added and the soln. diluted to 49 ml; $SnCl_2$ soln. [1·2% in HCl (1:5)] (1 ml) is added and the extinction is measured at 660 mμ. A precision of ± 2% is claimed in the range 50 to 150 μg of SiO_2.
T. R. Andrew

4707. **Analysis of used silicon - copper alloys [employed in the synthesis of chlorosilanes].** A. P. Terent'ev, B. M. Luskina and S. V. Syavtsillo. *Zavod. Lab.*, 1959, **25** (3), 288–289.—Determinations of C, Cl, Si, Cu and Fe on a single sample are described. Wet combustion of the sample (0·2 to 1·5 g) is carried out in a flask containing 10 ml of conc. H_2SO_4 and 3 to 4 ml of CrO_3 soln. (70 g of CrO_3 dissolved in 100 ml of water) in a current of O. The oxidation products pass through a heated (700° to 750°) quartz tube containing pumice which has been soaked in 10% $(NH_4)_2Cr_2O_7$ soln. and ignited. The absorption of Cl and HCl takes place in a vessel containing hydrazine hydrate and acetic acid at pH 6 (a soln. that does not absorb CO_2), and CO_2 is absorbed in soda asbestos. The contents of the reaction flask are heated with a few drops of methanol or ethanol together with 40 to 50 ml of water, and the insoluble matter (free Si with small amounts of SiO_2, SiC, etc.) is filtered off, washed, and dried to const. wt. at 110° to 130°. In the filtrate Cu and Fe are determined by known methods.
G. S. Smith

4708. **Use of naphthalene derivatives in inorganic analysis. VII. The nitronaphthols as fluorimetric reagents for stannous tin.** J. R. A. Anderson, A. J. Costoulas and J. L. Garnett (N.S.W. Univ. of Technol., Sydney, Australia). *Anal. Chim. Acta*, 1959, **20** (3), 236–242.—Thirteen nitro derivatives of 1-naphthol and 2-naphthol are listed that can be used for the fluorimetric detection of Sn^{II} in concn. as low as 10^{-4} to 10^{-5} g. The 2-nitro compounds give a more intense fluorescence than do the 1-nitro compounds; 8-nitro-1-naphthol is least reactive. *Procedure*—Place a drop of the sample soln. on Whatman No. 1 chromatographic paper and spray the air-dried spot with a 0·1% soln. of the reagent in a mixture (1:1, by vol.) of 96% ethanol and 10 *N* HCl. Then spray the dry paper with 15 *N* aq. NH_3, again air-dry and view under u.v. light. The fluorescence is specific for Sn^{2+}.
W. J. Baker

4709. **Determination of lead by the anodic amperometric method.** V. A. Khadeev and A. G. Nikurashina (V. I. Lenin Central Asia State Univ.). *Zavod. Lab.*, 1959, **25** (3), 283–285.—The method is

based on the current due to the oxidation of Pb^{2+} to PbO_2 at a rotating platinum electrode, and titration is carried out with $K_2Cr_2O_7$ soln. at a potential of 1 V *vs.* the S.C.E. in the presence of an acetate buffer soln. of pH ≃ 4. With contents of Pb < 0·2 mg per ml, the soln. should contain 10 to 15% of ethanol. No interference is caused by Mn and Ni at concn. ⩾ twice the concn. of Pb. Even small amounts of Co interfere. The method is applied to the determination of Pb (4·33%) in leaded bronze. An aliquot (25 ml) of the soln. of the alloy is boiled and treated with 0·5 ml of 1·5 *M* acetate buffer soln., then cooled and titrated. G. S. SMITH

4710. **Determination of lead in plating electrolytes, using ethylenediaminetetra-acetic acid (disodium salt) and Xylenol orange indicator.** K. E. Langford. *Electroplating*, 1959, **12** (3), 103, 119.—From a study of the volumetric determination of Pb, the following method is recommended. Dilute 1 ml of test soln. to 400 ml with water, add 0·5 to 1 g of hydroxyammonium chloride, 6 to 8 drops of a 1% aq. soln. of Xylenol orange and 1 g of hexamine, and titrate with 0·2 *N* EDTA (disodium salt) to a pale-yellow end-point. The titration is accurate to within 0·05 ml.

C. H. COWPER-COLES

4711. **Spectrographic determination of small quantities of antimony and tin in lead after a preliminary concentration by co-precipitation.** J. Downarowicz and Z. Zagórski (Anal. Lab., Central Lab. of Accumulators and Cells, Poznan, Poland). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 445–454.—Small amounts of Sb and Sn in lead can be determined spectrographically after co-pptn. with MnO_2 as collector; an almost 200-fold concentration is achieved, thus permitting the determination of 0·001% of Sb and 0·0005% of Sn. The method is designed for a quartz spectrograph of medium dispersion and the samples are excited by means of an a.c. arc between carbon electrodes. The manganese spectrum served as reference spectrum and the following line-pairs were used—for Sb and Sn from 0·01 to 0·1%, Sb 2878 A or Sn 2863 A and Mn 2915 A; for Sb from 0·001 to 0·01%, Sb 2311 A, Mn 2305 A; for Sn from 0·0005 to 0·01%, Sn 2355 A, Mn 2473 A. The relative error is ⩾ 12% for Sb and 20% for Sn. W. B. MIASKOWSKI

4712. **Rapid volumetric method for determining titanium in ferrotitanium.** E. F. Konstantinov (Stalin Metallurgical Works). *Zavod. Lab.*, 1959, **25** (2), 134–136.—The alloy is dissolved in dil. H_2SO_4 in an atmosphere of CO_2 and the Ti^{3+} are titrated with a soln. of Fe^{3+}. Since Ti^{4+} are not formed no special reduction stage is required.

G. S. SMITH

4713. **Determination of small amounts of titanium in tin alloys.** O. I. Popova (Inst. of Metal Ceramics and Special Alloys, Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (2), 148.—The sample (0·5 to 1 g) is dissolved in 10 to 15 ml of conc. H_2SO_4 and the cooled soln. is treated with 0·5 ml of 1% chromotropic acid soln. and diluted to 25 ml with conc. H_2SO_4. The colour intensity is compared with that of a scale of standards. No interference is caused by Sn, Nb and Ta. The method is applicable to contents of Ti between 0·001 and 0·007%.

G. S. SMITH

4714. **Polarographic determination of q valent titanium in the presence of foreign i orthophosphoric acid as electrolyte.** P. Sz L. Máczay and A. Gergely (Inst. for Inor Anal. Chem., L. Kossuth Sci. Univ., Deb Hungary). *Magyar Kém. Foly.*, 1959, **65** (2 72.—The polarographic determination of T 2 *M* H_3PO_4 is described: $E_{\frac{1}{2}} = -0{\cdot}229$ V. polarogram is not influenced by a 100-fold of Cd^{II}, Co^{II} or Ni^{II}, or a 10-fold excess of If these ions are present together, a 20-fold of Cd^{II}, Co^{II} and Ni^{II}, or a 10-fold excess these plus Mn^{II} does not interfere; Fe^{III}, V^{V} or W^{VI} in an amount equal to that of Ti^{I} not interfere, but very small amounts of Mo

A. G. F

4715. **Application of infra-red spectrosco the analysis of titanium tetrachloride.** Tsekhovol'skaya, T. A. Zavaritskaya, G. S. D and V. M. Chulanovskiĭ (All-Union Alum Magnesium Inst.). *Zavod. Lab.*, 1959, **25** (3) 302.—The determination of HCl (at 28 2929 cm^{-1}), of $TiOCl_2$ (at 821), of $VOCl_3$ (at of $COCl_2$ (at 1654 or 1812), of hexachlorob (at 1301), of dichloroacetyl chloride (at 1 1809), of trichloroacetyl chloride (at 180 chloroacetyl chloride (at 1785 or 1820), a CO_2 (at 2338) is described. The method c used for determining the solubility of CO_2 $COCl_2$ and hexachlorobenzene in $TiCl_4$. I presence of excess of $TiCl_4$, hydrolysis takes in accordance with the equation—

$$TiCl_4 + H_2O = TiOCl_2 + 2\,HCl.$$

G. S. SM

4716. **Analytical applications of Xylenol o I. Determination of traces of zirconium.** Cheng (Metals Div., Kelsey-Hayes Co., N.Y., U.S.A.). *Talanta*, 1959, **2** (1), 61 Zirconium (5 to 60 μg) can be determin measuring the extinction of the complex f with Xylenol orange in 0·2 *N* H_2SO_4. *Proce* To the neutral sample soln. add H_2SO_4 (2 ml) or $HClO_4$ (5 *N*) (4 ml) and aq. Xylenol soln. (0·05%) (2 ml). Dilute to 25 ml and m the extinction at 535 mμ. Niobium, Hf, M Fe^{III} and Sn^{II} interfere, but the effect of N and Sn^{II} can be eliminated by carrying o test in 0·3 to 0·5 *N* H_2SO_4; Fe^{III} can be r with ascorbic acid and Bi with Cl^-. Fl EDTA, nitrilotriacetic acid, citrate, ta oxalate, thiosulphate and phosphate also int

W. T. CAR

4717. **Volumetric determination of zirc II. The oxidation of zirconium tetramar with chromic acid.** A. Schneer and H. Har (Inst. of Gen. and Inorg. Chem., L. Eötv Univ., Budapest). *Magyar Kém. Foly.*, 19 (2), 64–69.—Mandelic acid selectively preci Zr in HCl soln. Collect the tetramandelate, d it in conc. H_2SO_4 and add it to 0·25 *N* Na_2C conc. H_2SO_4. Add sufficient water to m 80 to 90% (w/w) with respect to H_2SO_4, a 0·06 *N* with respect to $Na_2Cr_2O_7$; heat in an a at 115° for 20 min. and back-titrate the ex $Na_2Cr_2O_7$ with $FeSO_4$ soln. with ferroin as ind 1 ml of 0·1 *N* $Na_2Cr_2O_7 \equiv 67{\cdot}07$ μg of Zr. 0·5 to 5 mg of Zr is present, a + 2% correcti give an accuracy of ± 1% but, if only 0·5 mg of Zr is present, the error may be ± The method can be used for rocks con 0·01 to 0·1% of Zr. Compounds form hydrated oxide in acid soln. do not interfere

natometric determination of mandelic acid is lescribed. A. G. Peto

18. **Rapid complexometric determination of nium in zirconium glass and concentrates.** Panasyuk and N. A. Miroevskaya (All-Union Res. Inst. of Glass Fibre). *Zavod. Lab.*, 1959, 2), 147.—A simplified modification of the od of Goryushina and Romanova (*cf. Anal.* ., 1958, **5**, 1484) is presented. With con- ates of Zr, technical dioxide or zirconium the sample (0·2 g) is fused in a silver crucible a mixture of 5 g of KOH and 2 g of $Na_2B_4O_7$ he aq. soln. of the melt, together with insoluble er, is treated with 50 ml of conc. HCl and the e diluted to 250 ml. An aliquot (25 ml) is d with 25 ml of 2 *N* HCl and 50 ml of water titrated with 0·05 *N* EDTA (disodium salt) to hrome black T, the final end-point being ob- d after further heating. G. S. Smith

19. **Complexometric determination of zirconium orides and nitrides.** E. I. Nikitina. *Zavod.* , 1959, **25** (2), 142.—With borides, 0·2 g is lved in 20 ml of dil. H_2SO_4 (1 : 1) and 5 ml of . HNO_3, oxides of N are boiled off, the in- ble matter (carbide) is separated and fused $K_2S_2O_7$, and the aq. soln. of the melt is com- d with the main soln., which is then diluted 00 ml. With nitrides, 0·2 g is dissolved in l of conc. H_2SO_4, the soln. is evaporated to ng, and the cooled soln. is diluted to 100 ml. the titration, 50 ml is mixed with an accurately sured amount (10 to 25 ml) of 0·1 *N* EDTA dium salt), 20 ml of 20% tartaric acid soln., of ammonium acetate soln. (concn. not stated) 2 drops of Tropaeolin OO (C.I. Acid Orange 5) cator soln., and treated with aq. NH_3 to give a pink colour. The soln. is boiled for 10 min. lissolve a pptd. complex of Zr and EDTA), then ed and mixed with 1 g of thiourea, and titrated a soln. of $Bi(NO_3)_3$ to the appearance of a ht yellow colour. G. S. Smith

20. **[Determination of] thorium in stone meteor- by neutron activation analysis.** G. L. Bate, R. Huizenga and H. A. Potratz (Argonne Nat. ., Lemont, Illinois). *Geochim. et Cosmoch.* , 1959, **16** (1–3), 88–100.—Full details are given the preparation and slow neutron activation of samples, during which ^{232}Th by slow neutron orption produces ^{233}Th which decays with a nin. half-life to ^{233}Pa. Radiochemical assays based on ^{233}Pa because of the latter's longer -life and its intense γ-radiation of 310-keV gy which permits convenient detection by tillation counting. The procedures for separa- of Pa are based on its co-deposition with certain ipitates (especially manganese dioxide and um fluorozirconate), extraction into appropriate nic solvents, and pronounced complexing lency with HF. Two essentially different ochemical procedures for Pa isolation, differing n in method of initial opening up of the samples in subsequent cycles of purification, are ribed in detail. R. A. Howie

21. **Determination of aluminium in thorium e and a separation scheme for interfering ions.** Goldstein, D. L. Manning and O. Menis (Oak ge Nat. Lab., Tenn., U.S.A.). *Talanta*, 1959,), 52–60.—Aluminium (0·5 to 150 μg) in impure ium oxide is isolated by removing Th, Fe and with thenoyltrifluoroacetone (**I**) and then extracting the Al with 8-hydroxyquinoline in $CHCl_3$ in the presence of H_2O_2, NaCN and nitrilo- triacetic acid. *Procedure*—Dissolve the sample (0·1 g) in boiling HNO_3 (1 : 1) (20 ml) containing a little HF. Add $HClO_4$ (72%) (3 ml) and evaporate to fumes. Cool, add H_2O (5 ml) and ammonium acetate soln. (50%) (8 ml) and adjust to pH 1·5 with $HClO_4$. Dilute to 25 ml with H_2O and extract twice with a 0·5 *M* soln. of **I** in *iso*butyl methyl ketone (10 ml) and wash once with the ketone (10 ml). Dilute the aq. phase to 75 ml, add nitrilotriacetic acid soln. (0·2 *M*) (5 ml), H_2O_2 soln. (3%) (2 ml), acetate buffer (200 g of ammonium acetate and 70 ml of conc. aq. NH_3 in 1 litre) (2 ml) and 8-hydroxyquinoline soln. (2%) (2 ml). Adjust to pH 8 with aq. NH_3 (1 : 1) and extract twice with $CHCl_3$ (20 ml). Combine the $CHCl_3$ extracts, dilute to 50 ml with $CHCl_3$ and dry with anhyd. Na_2SO_4. For $\geq$ 10 μg of Al measure the fluorescence intensity in a spectrophotometer with a fluorescence attachment. For 10 to 150 μg, measure the extinction at 390 mμ. Slightly modi- fied procedures are necessary for samples containing Ni, Zn, Cd, Mn and Co. The spectrophotometric method gives a coeff. of variation of $\simeq \pm 3\%$ and the fluorescence method $\simeq \pm 5\%$.

W. T. Carter

4722. **Effect of the mode of combustion on nitrogen determination by Kjeldahl's method.** L. Rozental (Med. Acad., Warsaw). *Roczn. Zakl. Hig., Warszawa*, 1958, **9**, 183–197.—Of the catalysts studied (HgO, selenium metal and $CuSO_4.5H_2O$), yellow HgO proved to be the best. Mixed catalysts saved some combustion time, but the values obtained were lower than for HgO alone. The addition of oxidising agents resulted in a further loss of N. The optimum concn. of K_2SO_4 was found to be 7·5 g per 20 ml of H_2SO_4 and that of HgO was found to be 1 g per flask.

Chem. Abstr.

4723. **Chromatographic separation and micro- analysis of nitrogen and nitric oxide in mixtures of nitrogen and oxides of nitrogen using an adsorption column.** M. Lefort and X. Tarrago (Lab. de Phys. Nucléaire, Fac. des Sci. de Paris à Orsay, France). *J. Chromatography*, 1959, **2** (2), 218–220 (in French).—Oxygen, N and NO can be separated on a 3·2-metre column of Linde Molecular Sieve 5A at 84°, with H as carrier gas; 0·010 to 0·500 ml of O or N can be determined with a precision of $\pm$ 0·004 ml. Nitric oxide is not com- pletely eluted and the column must be conditioned with NO before use in order to obtain quant. results. For this component the precision is $\pm$ 0·020 ml in the range 0·050 to 0·150 ml, and $\pm$ 0·006 ml in the range 0·150 to 0·500 ml; N_2O does not interfere. W. T. Carter

4724. **Determination of water and aniline in hydrazine by infra-red spectrometry.** F. Lalanne (Office Nat. Ind. de l'Azote). *Chim. Anal.*, 1959, **41** (2), 45–48.—Preliminary investigations were carried out on the analysis of hydrazine - H_2O mixtures by the method of Cordes and Tait (*Anal. Chem.*, 1957, **29**, 485) and the analysis of hydrazine - aniline mixtures by differential absorption between 6·4 and 7·4 μ. It is concluded that the presence of up to 2% of aniline leads to an error of 0·4% in the determination of H_2O by the Cordes and Tait method; that the extinction at 6·66 μ varies with the H_2O content of hydrazine - aniline mixtures from 1 to 6% of H_2O, but the difference between the extinctions of the max. at 6·66 μ and the

min. at 6·5 μ is independent of the H_2O content for a given aniline content; and that the extinction of the min. at 7·15 μ is proportional to the H_2O content for values $< 7\%$. Two methods have been developed for the complete analysis of ternary hydrazine mixtures containing $< 10\%$ of H_2O and aniline. (*i*) Aniline is determined by measuring the difference in the extinctions at 6·5 μ and 6·66 μ, and H_2O by the Cordes and Tait method with the addition of a correction of 0·2% for each 1% of aniline. The method gives an absolute accuracy of 0·05% for aniline and 0·1% for H_2O in the concn. range 0 to 2·5% of aniline. Disadvantages are that two series of measurements and relatively large samples are necessary. (*ii*) Aniline and H_2O are determined simultaneously by measuring the differential absorption curves of the mixture against pure hydrazine between 6·4 and 7·4 μ, the H_2O content being determined by the absorption at 7·15 μ and the aniline content as in method (*i*). The accuracy is 0·05% for both aniline and H_2O for the concn. range 0 to 2·5% of aniline and 0 to 7% of H_2O. S. M. MARSH

4725. **Colorimetric determination of nitrite with NNN′N′-tetramethylbenzidine.** M. Matrka and F. Navrátil (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1959, **9** (2), 75–76.—NNN′N′-Tetramethylbenzidine (**I**) reacts with nitrite in an acid medium yielding the orange NN′-dimethyldiphenoquinonedi-imine dimetho-chloride which can be determined photometrically. *Calibration curve*—To the soln. of **I** (0·1% in *N* HCl) (1 ml) add *N* HCl (9 ml) and 0·5 to 3 ml of aq. nitrite soln. (0·01 g of $NaNO_2$ per litre). After 15 min. dilute to 100 ml and after 3 hr. measure the extinction at 475 mμ. The mechanism of the reaction has been studied by means of the colorimetric titration of 10^{-4} *M* **I** with 10^{-3} *M* $NaNO_2$. Dichromate, MnO_4^-, Ce^{4+}, SO_3^{2-} and $S_2O_4^{2-}$ interfere. This reaction can also be used for the colorimetric determination of strongly oxidising agents (*e.g.*, Cl, Br and chromates). J. ZÝKA

4726. **Rapid determination of phosphorus in coal and coke.** I. I. Édel'man, L. S. Zabora and N. D. Khizhnyak (Ukraine Sci. Res. Coal Chemistry Inst.). *Zavod. Lab.*, 1959, **25** (2), 159–160.—The sample (1 g of coal or 0·5 g of coke) is ashed at 850° with free access of air and the residue is dissolved in 10 ml of a mixture of H_2SO_4 and HNO_3 (2:1). Two aliquots (25 ml) of the filtered and diluted soln. (100 ml) are treated at 50° to 60° with 4 ml of a soln. of Fe^{3+} [108 g of $(NH_4)_2SO_4.Fe_2(SO_4)_3$ in 300 to 350 ml of water] and 5 ml of conc. HCl. Each soln. is diluted to 500 ml and treated with aq. NH_3 and 4 *N* HCl so that there is a 2-ml excess of the acid. The soln. are boiled for 1 min. with 5 ml of 20% Na_2SO_3 to decolorise. More Na_2SO_3 is added if necessary to reduce all the Fe^{3+}. If a gold-coloured tint appears, a few drops of 4 *N* HCl are added. The cooled soln. are then treated with 6 ml of 4 *N* HCl and one of them is treated with 4 ml of 5% $(NH_4)_2MoO_4$ soln., the other soln. serving as a control. The colour intensity is measured with a red filter, and the content of P is calculated from a calibration curve. G. S. SMITH

4727. **Examination of oxy acids of phosphorus by electrochromatography.** Takuya R. Sato (Argonne National Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 841–845.—The extension of the distance of differential electrical migration by the use of filter-paper moistened with 0 acetic acid permits the separation of slow mig in mixtures of complex and condensed phosph oxy acids. The separation is also aided by varia in the electrolyte and by the addition of rea (*e.g.*, Zn^{2+}) in zones retaining certain compo only. The d.c. potential is either 5 or 10 to per cm (for 17 to 20 and 2 to 4 hr., respectiv The resolved zones are located by spraying molybdate, neutron activation (and scanning autoradiography. The sequence and separat are established for Na_2HPO_4, Na_2HPO_3, NaH_2 $Na_4P_2O_7$, $Na_5P_3O_{10}$, $(NaPO_3)_3$, $(NaPO_3)_4$, Na_3H $Na_3HP_2O_5$, $Na_2H_2P_2O_5$ and $Na_2H_2P_2O_6$. The s tivity of the method is very high (approx. 1 for non-radioactive phosphorus compounds). method is suitable for the examination of irrad natural products and the purity and deterior of standard prep., and for the purification of tra J. P. STE

4728. **Simultaneous titrations of antimony arsenic with the dead-stop end-point.** Nan Young, T'sing-Chuan Tien and Sheau-Shya (Chem. Dept., Peking Univ.). *Acta Chim. Si* 1959, **25** (2), 106–109.—The dead-stop end-p method is applied to the determination of Sb As in anode mud without previous separa standard $Ce(SO_4)_2$ soln. being used as the tit The sample is dissolved in conc. H_2SO_4. digest is diluted with water and adjusted to in HCl; Sb^{5+} are titrated with 0·1 *M* $Ce(SO_4$ an applied voltage of 60 mV at 0°, at which t the Sb^{3+} - Sb^{5+} system is reversible. The sol then adjusted to 4 *N* in HCl and warmed to 1 22°, ICl soln. being added to render the A As^{5+} system reversible. The titration is conti until As^{3+} are completely oxidised. A com analysis takes $\simeq$ 3 hr. Satisfactory results fo and As in two samples of anode mud are repo S. H. YU

4729. **New colorimetric method for the d mination of tervalent antimony in the presen tervalent arsenic.** D. G. Constantinescu, Baiulescu and R. Oteleanu (Parhon Univ.). *R.P.R., Stud. Cercet. Chim.*, 1959, **6** (3), 439–4 The method is based on the reaction of a metha soln. of antimony and arsenic chlorides wi methanolic soln. of quercetin (0·05%), the resu yellow colour being measured in the 1-cm cell Pulfrich photometer with a S42 filter. From to 10·98 μg of Sb per ml can be determi Arsenic does not interfere unless its concn. is than six times that of the Sb. H. SH

4730. **Rapid methods of determining anti in bronze.** R. S. Volodarskaya. *Zavod. Lab.*, **25** (2), 143–144.—Methods based on the iodide the thiourea complexes are described. With former, 0·1 g of sample is heated with 5 m water and 10 ml of conc. H_2SO_4 and the c soln. is mixed with 50 ml of water and then dil to 100 ml with dil. H_2SO_4 (1:5). An aliquot (1 is mixed with 30 ml of dil. H_2SO_4 (1:5), 10 m 10% thiourea soln. and 20 ml of 10% KI and then diluted to 100 ml with dil. H_2SO_4 (After 8 to 10 min. the extinction is measured a violet or blue filter, and the content of S found by means of a calibration curve. With thiourea method, 0·1 g is dissolved in 5 m water and 10 ml of conc. H_2SO_4, and the sol evaporated to fuming, then diluted with v (20 ml), treated with 60 ml of 10% thiourea s and diluted to 100 ml with dil. H_2SO_4 (1:5).

tion is measured after 5 to 7 min., but within n., after the addition of thiourea. A violet or ilter is used. The methods are suitable for nts of Sb from 0·1 to 7%. G. S. Smith

1. **Spectrographic analysis of antimonate, te and products obtained during their treat-** I. G. Yudelevich, F. P. Polatbekov and Kovaleva (S. M. Kirov Kazakh State Univ.). *Lab.*, 1959, **25** (3), 305–307.—Conditions for ectrographic determination of Sn, Cu, Pb, Fe, l, Te and In in antimonate, and of Sb, Fe, s, Pb, Te and In in stannate, and for the sis of metallic tin and antimony, etc., are bed. G. S. Smith

2. **Spectrographic analysis of antimony and ony sulphide for heavy metals.** G. F. G. (Res. Dept., African Explosives & Chem. tries, Ltd., P.O. Northrand, Transvaal). *ochim. Acta*, 1959, (1), 49–55.—A spectro-ic method is described for the determination Cu, Fe and Pb in the range 0·01 to 1·0% in lic antimony. The sample is first converted the oxide by treatment with conc. HNO_3, compared with synthetic standards by arcing mpletion in an 8-amp. d.c. arc. The same nts, together with Bi and Zn, are determined timony sulphide by a similar technique. ra are evaluated by means of a "Respektra" ating board; coeff. of variation between 5 0% are claimed. P. T. Beale

3. **Photometric determination of bismuth tetraphenylphosphonium bromide.** Nobusiha no and Akira Kawase. *Trans. Nat. Res. Met., Japan*, 1959, **1** (1), 69–71 (in English).—id method for determining Bi is based on the of orange tetraphenylphosphonium tetra-ismuthate, which is sol. in $CHCl_3$. *Procedure*—e sample soln. (⩾120 μg of Bi) are added 3 ml % KI soln., 5 drops of phenylhydrazine soln. n dil. H_2SO_4) and 3 ml of 0·01 *M* aq. tetra-rlphosphonium bromide. The mixture, which l be ≃2 *N* in H_2SO_4, is set aside for 10 min., hen extracted with $CHCl_3$. After separation, rganic layer is filtered through a cotton pad a 1-cm cell, and its extinction is measured st that of a $CHCl_3$ blank at 505 mμ. Inter-ce is shown by Fe^{3+}, Cu^{2+}, Hg^{2+}, Cd^{2+}, Sn^{2+} Sn^{4+}; that due to Sb^{3+} is eliminated by the on of K Na tartrate. Beer's law is obeyed up 0 μg of Bi. J. H. Waton

4. **Complexometric method of determining th in multi-component alloys [of bismuth, cadmium and tin].** A. I. Cherkesov and A. S. ikova (Astrakhan Tech. Inst. of the Fishing try). *Zavod. Lab.*, 1959, **25** (2), 140–141.—lloy (0·2 to 0·4 g) is dissolved in 10 to 15 ml HNO_3 (1:1) and the cooled soln. is diluted 0 ml. An aliquot (10 ml) of the well-shaken (some of the Bi is adsorbed on the insoluble tannic acid) is treated with 5 drops of a 1% olic soln. of haematoxylin, and titrated with *M* EDTA until the colour changes from to pale yellow. The soln. is then vigorously n, the violet colour reappearing, and the ion is continued. Shaking and titration are ued until all the Bi has been desorbed. If ample contains from 4 to 15% of Sb, the to be titrated is first mixed with 0·5 ml of ated NaF soln. Gallein can be used as indicator instead of haematoxylin, but the colour change at the end-point is somewhat less sharp. G. S. Smith

4735. **Spectrophotometric and photometric micro-determination of vanadium.** F. Bermejo Martínez and M. Paz Castro (Lab. of Anal. Chem., Fac. of Sci., Univ. of Santiago de Compostela, Spain). *Chemist Analyst*, 1959, **48** (1), 2–3.—Acid soln. of VO^{2+} give an intense colour with H_2O_2 and 1:2-di-(2-aminoethoxy)ethane-NN′-tetra-acetic acid (EGTA) (**I**). The test soln. (⩾120 μg of V) is treated with 2 ml of 5% aq. soln. of **I** (disodium salt), 10 ml of Clark and Lubs buffer soln. (pH 1·70) and 1 ml of 30% H_2O_2 soln. The pH is adjusted to 1·70 (pH meter) and the mixture diluted to 25 ml. After 25 min. the absorption is read spectrophotometrically at 426 mμ or photometrically with an Ilford No. 601 filter, in a 1-cm cell, and results are referred to the appropriate calibration curve. Very high concentrations of Fe^{III}, Ti^{III}, Cu^{II}, U^{VI}, Mo^{VI} and W^{VI} interfere, but moderate amounts of Fe and Ti can be masked with fluoride. Moderate amounts of other metals, minor amounts of W^{VI}, and SO_4^{2-}, PO_4^{3-}, NO_3^-, Br^-, Cl^-, NH_4^+, K^+ and Na^+ do not interfere, so that a variety of reagents can be used for dissolution of the sample and adjustment of pH. Owing to the limited interferences, microgram amounts of V can be determined in complex materials. R. E. Essery

4736. **Method for the spectrographic determination of trace impurities in niobium.** D. E. Fornwalt and M. K. Healy (Pratt & Whitney Aircraft Div., CANEL Operations, Middletown, Conn., U.S.A..) *Appl. Spectroscopy*, 1959, **13** (2), 38–40.—The method described involves the conversion of Nb into the oxide by heating in platinum at 800°. A 10-mg sample plus 10 mg of Li_2CO_3 as a spectrographic buffer and 25 μg of La as internal standard is excited in a 20-amp. d.c. arc. The spectrogram obtained can be used to determine Cd, Cr, Co, Fe, Mn, Mo, Ni, Sn and Ti in the 0·001 to 0·1% concn. range. A separate 20-mg sample is excited to determine B (0·0001 to 0·0015%), Al and Si (0·001 to 0·1%), Ta (0·025 to 1·5%) and Zr (0·001 to 1·5%). The coeff. of variation ranged from 2% for Zr to 19% for Sn. K. A. Proctor

4737. **Chemistry of niobium and tantalum. XIX. Separation of niobium and tantalum.** H. Schäfer (Anorg.-Chem. Inst. d. Univ., Münster, Westphalia, Germany). *Angew. Chem.*, 1959, **71** (4), 153–160.—A review is given of procedures for the separation of Ta and Nb, subdivided under three main headings —fractionation of compounds based on differences in physical properties; separations based on reduction of Nb^V; and separations based on differences in stability towards hydrolysis (including ion-exchange techniques). (134 references.) T. R. Andrew

4738. **Photometric determination of tantalum with phenylfluorone.** C. L. Luke (Bell Telephone Lab. Inc., Murray Hill, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 904–906.—Tantalum forms a red complex with 2:3:7-trihydroxy-9-phenyl-6-fluorone (**I**) suitable for photometry at 530 mμ. Interference by other metals is avoided by extracting Ta from an HF - HCl soln. with *iso*butyl methyl ketone (**II**) and masking any co-extracted metals with EDTA (disodium salt) (**III**). For satisfactory accuracy (not defined), the pH and the concn. of F^-, **I**, **III** and buffer must be closely controlled. *Procedure*—Dissolve the dry sample (free from

SO_4^{2-}, ClO_4^- or Cr^{VI}) in 5 ml of acid (5 ml of HF, 5 ml of HCl and 90 ml of H_2O), cool, and extract with **II** (2 × 5 ml). Separate and evaporate the organic phase. Add $HClO_4$ (0·25 ml) and HNO_3 (0·25 ml) and heat until $HClO_4$ is copiously evolved. Add HNO_3 (0·25 ml) and evaporate to dryness. Cool, add 1 ml of HF (1:99) and warm until dissolution is complete. Add 5 ml of 10% aq. **III**, 10 ml of ammonium acetate - acetic acid buffer (pH 4·5) and 10 ml of 0·01% **I** in 10% methanol, and set aside for 30 min. Add 5 ml of satd. H_3BO_3 soln. (corrosion inhibitor), make up to 50 ml with acetic acid (1:9), and immediately determine the extinction at 530 mμ. Read the Ta content from a calibration curve. J. P. STERN

4739. **Spectrographic determination of micro amounts of titanium, niobium and tantalum.** T. M. Moroshkina and V. K. Prokof'ev. *Vestn. Leningr. Univ.*, 1959, No. 10, *Ser. Fiz. i Khim.*, (2), 143–148.—The following lines were used in the determinations—Ti 4305·92 A, Nb 2927·81 A, Ta 2933·55 A, Nb 2950·88 A, and Ta 2951·9 A. Uranium (4297·11 A) or Cr (4297·73 A) was used as an internal standard in the determination of Ti, Ta in the determination of Nb, and Nb in the determination of Ta; Fe (2929·01 A) was used as the comparison element in the determination of Nb and Ta. Spectra were excited in a stabilised a.c. arc at 220 V and a current of 7 to 8 amp. for the determination of Ti and a current of 20 amp. for Ta and Nb. The admixture (prepared by fusing the oxide of Ti, Nb or Ta with $K_2S_2O_7$ and dissolving the product in conc. H_2SO_4) and the internal standard soln. are added to the sample, which is ignited at 700° for 20 to 25 min. and then placed in the bore of a carbon electrode. The results showed that the direct spectrographic determination of small amounts of Ti in natural samples of complex composition with evaporation of the sample from a bead and with the admixture method presents no difficulties; Ti was determined in the range 5 × 10^{-4} to 0·5% with an accuracy of ±10%. The simultaneous determination of Nb and Ta can be carried out for a range of 0·01 to 1%; with lower concn. of Nb and Ta, the determination can be carried out with ratios of Nb to Ta of 1:1 to 1:2. K. R. C.

4740. **Paper electrophoresis of protactinium(V).** J. Vernois (Inst. du Radium, Paris, France). *J. Chromatography*, 1959, **2** (2), 155–161 (in French).—The behaviour of Pa^V, Fe^{III}, U^{VI}, Pb^{IV}, Bi^{III}, Zr^{IV}, Ta^V, Nb^V, Ti^{IV}, Th^{IV} and Po^{III} has been studied with HF and mixtures of HCl and HF as electrolytes and a potential difference of 150 V. Protactinium can be separated from Zr, Ti, Th and Fe, and Zr can also be separated from Nb. W. T. CARTER

4741. **Separate determination of peroxide oxygen and superoxide oxygen in superoxides.** I. V. Vol'nov and E. I. Latysheva (Kurnakov Inst. Gen. and Inorg. Chem., Acad. Sci., Moscow). *Zhur. Anal. Khim.*, 1959, **14** (2), 242–243.—In the analysis of materials containing peroxides and superoxides, the gasometric determination of peroxide oxygen can be satisfactorily replaced by a permanganate titration (0·1 *N* $KMnO_4$) of the peroxide oxygen formed in acetic acid soln. during the gasometric determination of superoxide oxygen by the method of Seyb and Kleinberg (*Anal. Chem.*, 1951, **23**, 115); the presence of diethyl phthalate in the soln. does not affect the titration. K. R. C.

4742. **Determination of small amounts of h gen in sulphur. Application to the determi of water in the presence of sulphur trioxide** Barrier (Lab. d'Essais des Prod. Chim., Com Saint-Gobain, Chauny et Cirey, Aubervi France). *Chim. Anal.*, 1959, **41** (3), 97–1 Different types of organic matter present in su are discussed and three methods for the mination of H are described. *Determination by combustion*—The SO_3 and SO_2 formed by bustion of S are absorbed on Na_2CO_3 - $CaSO_4$ the H_2O is absorbed with anhydrone. Appa and procedure are described. The method i subject to systematic error. If sulphur is rep with H_2SO_4, the H_2O content of the acid m similarly determined, but more precisely tha titration. *Determination of humidity in gases co ing SO_3*—A simple furnace is described for fixin as before. *Determination of H by thermal crack* The sample of sulphur is heated at the b.p. for under an inert gas, and the H_2S evolved is abs in Zn or Cd acetate soln.; the determinati completed iodimetrically. The method is and sufficiently accurate (80 to 90% recovery to serve for the control of S purification. Mo does not interfere. P. D. PARR-RICHA

4743. **Solubility of hydrogen sulphide in** R. Belcher and J. P. G. Farr (Dept. of C Univ., Edgbaston, Birmingham, England). *Ta* 1959, **2** (1), 95–97.—The H_2S generated from fe sulphide and HCl (1:1) contains large amou hydrogen. A generator which had been wo for several hours produced gas containing ≃ (v/v) of H_2S, but after shutting down for the concn. of H_2S fell to 50% (v/v). The pur the H_2S increases with increase in the ra generation. Only rarely were soln. 0·05 *M* i produced at room temp. under conditions parable to those existing in qual. analysis. must be taken into account in mass-action lations concerned with the pptn. of sulphides. W. T. CAR

4744. **The sulphur acids. XVI. Iodimetri colorimetric determination of sulphanes [hyd polysulphides], elementary sulphur and sulph sulphur mixtures.** M. Schmidt and G. T (Inst. f. Anorg. Chem. d. Univ., München) *anal. Chem.*, 1959, **166** (4), 274–283.—Hyd polysulphides (H_2S_x) react with Na_2SO_3 to H_2S and $Na_2S_2O_3$, according to the equat $H_2S_x + (x-1)SO_3^{2-} \rightarrow H_2S + (x-1)S$ The H_2S is absorbed in a soln. of Zn^{2+} or Cd^2 titrated, and, after addition of formaldehy react with the excess of SO_3^{2-}, the $S_2O_3^2$ determined. In homogeneous soln. S ma determined from the reaction $S_8 + 8SO_3$ $8S_2O_3^{2-}$. Details of the procedure for polysulp sulphur and for mixtures are given. Photo procedures have been developed based o formation of SCN^-, by reaction with CN^- measurement of the colour produced with A scheme of application for inorganic su compounds that can be converted into sulphi sulphur, and for organic sulphur compou given. T. R. AND

4745. **Determination of inorganic sulphur pounds in sulphate black liquor. I. Determi of thiosulphate and sulphite by potentio titration with mercuric chloride.** E. Bilberg Norwegian Pulp and Paper Res. Inst., *Norsk Skogind.*, 1958, **12** (11), 470–47

n).—Sulphite and $S_2O_3^{2-}$ can be titrated iometrically in the presence of S^{2-} which wave well separated from that due to $S_2O_3^{2-}$. te, CO_3^{2-}, PO_4^{3-} and acetate do not inter- $^-$ and SCN^- must be absent; Cl^- affect the ess of the end-point. A mercury pool con- through a platinum electrode is used as the or electrode; potentials are measured *vs.* the *Procedure for sulphite plus thiosulphate*— n aliquot containing 0·025 to 0·25 g of $Na_2S_2O_3$ 02 to 0·2 g of Na_2SO_3 to the titration beaker lute to 100 ml with air-free H_2O. Add 5 ml cerol and adjust the pH to between 7·0 and Titrate with 0·05 *M* $HgCl_2$. *Procedure for phate*—Make another aliquot up to 100 ml, the pH to between 7·0 and 7·5, add 5 ml of ormaldehyde soln. and set aside for 5 min. titrating. For the determination of SO_3^{2-} O_3^{2-} in black liquor, it is better to precipitate th a suspension of $CdCO_3$, filter off the CdS trate under N. The precision for the SO_3^{2-} ination is less than that of iodimetry; that O_3^{2-} is as good. P. D. PARR-RICHARD

6. **Analytical applications of oxidations with anide catalysed with osmium tetroxide. III. aneous determination of sulphur compounds.** lymosi and A. Varga (Inst. for Inorg. and Chem., Sci. Univ. of Szeged, Hungary). *ar Kém. Foly.*, 1959, **65** (2), 52–55.—For multaneous determination of $S_2O_4^{2-}$ and SO_3^{2-} O_4^{2-} and $S_2O_3^{2-}$, oxidise $S_2O_4^{2-}$ to SO_3^{2-} with $Fe)_6^{3-}$ (**I**) in a 0·5 to 1 *N* NaOH soln. After d-point, increase the alkalinity to 4 to 4·5 *N*, SO_4 soln. (**II**) (2 to 3 drops), heat at 50° to 60°, omplete the titration to oxidise SO_3^{2-} and to SO_4^{2-}. The first titration (*a* ml) gives the of $S_2O_4^{2-}$ directly; from the second (*b* ml) f SO_3^{2-} or $S_2O_3^{2-}$ can be calculated as ($b - 2a$). s the titrations are rapid, a nitrogen atmo- e is recommended; the end-point can be mined by the dead-stop method or potentio- cally. To determine $S_2O_4^{2-}$, SO_3^{2-} and , titrate $S_2O_4^{2-}$ in a 0·5 to 1 *N* NaOH soln. ; then determine SO_3^{2-} plus $S_2O_3^{2-}$ at the pH as above (*b* ml). Oxidise the next ot with an excess of iodine in weakly acid to SO_4^{2-} and $S_4O_6^{2-}$; remove the excess e with SO_3^{2-} and titrate the $S_4O_6^{2-}$ (which are d from $S_2O_3^{2-}$) in 5 *N* NaOH with **I** in the ice of **II** at 50° (*c* ml). All end-points are mined by the dead-stop method. From the titrations, the concn. can be calculated— of 0·1 *M* **I** ≡ 8·70 mg of $Na_2S_2O_4$ (from titra- *a*) or 6·30 mg of Na_2SO_3 (from titration *b*) or ng of $Na_2S_2O_3$ (from *c*). The accuracy is n 0·4%. The simultaneous determination of and $S_2O_3^{2-}$ is accomplished by oxidising the aliquot in 4 to 4·5 *N* NaOH at 50° with **I** in the presence of **II** and the second aliquot iodine as already described (*e* ml). The concn. $_3^{2-}$ is calculated from ($d - 8e/7$) and that of $^-$ is $8e/7$. To determine $S_2O_3^{2-}$ and $S_4O_6^{2-}$ taneously, oxidise both to SO_4^{2-} with **I** in NaOH (*f* ml) in the presence of **II**. Oxidise cond aliquot with iodine in weak acid; $S_2O_3^{2-}$ $S_4O_6^{2-}$; titrate this with **I** (*g* ml). The wt. of $^-$ is $f - g/8$ and that of $S_4O_6^{2-}$ is $f - (f -$ A. G. PETO

7. **Colorimetric determination of thiosulphate e presence of polythionates and hydrogen ite.** N.-H. Schöön (Dept. of Engng Chem., ners Tekniska Högskola, Göteborg, Sweden). *Acta Chem. Scand.*, 1959, **13** (3), 525–532 (in English).—The AgSCN method of Schöön (*cf. Anal. Abstr.*, 1959, **6**, 2117) gives erroneous results in the presence of polythionates and H_2SO_3. A new but less sensitive method is described in which thiosulphate is caused to react with *p*-benzoquinone in acetic acid soln., and the resulting colour is measured absorptiometrically. Polythionates do not interfere and interference from H_2SO_3 can be completely suppressed by the addition of excess of formaldehyde. The method is applicable to concn. of $S_2O_3^{2-}$ in the range 0·03 to 0·3 m*M*. W. T. CARTER

4748. **Plasmoquin [pamaquin] as a reagent in inorganic analysis. I. Detection of chromium(VI).** Te-Fung Lee, Nai-Ching Wang and Nai-Kuei Shen. *Acta Chim. Sinica*, 1958, **24** (6), 403–406.—Pamaquin in the presence of 0·1 *N* $HClO_4$ and 8% oxalic acid reacts with CrO_4^{2-} (or $Cr_2O_7^{2-}$) to give a red - violet colouration which is not extracted by ordinary organic solvents. The limit of identification is 0·05 μg at a dilution limit of 1 in 1×10^6. Quadrivalent Ce, Au^{3+}, VO_3^-, BrO_3^-, IO_3^-, IO_4^- and NO_2^- interfere with the detection of CrO_4^{2-}. Of the interfering ions, Ce^{4+} and Au^{3+} can be separated by aq. NH_3, and VO_3^- by 8-hydroxyquinoline - $CHCl_3$ extraction. In a mixture containing Ce^{4+}, Au^{3+}, VO_3^- and Cr^{3+} in concn. up to 20:20:20:1, Cr^{3+} (0·009 μg per 0·05 ml) can still be detected. Chromium should be previously oxidised in H_2SO_4 soln. with $(NH_4)_2S_2O_8$ or in alkaline soln. with H_2O_2 before separation of the ions. SCI. ABSTR. CHINA

4749. **New method for determination of molybdenum.** F. da Silva and L. de Moura (Inst. Sup. Técnico, Lisbon, Portugal). *Técnica*, 1959, **34** 437–443.—Conditions for the colorimetric determination of Mo with diphenylcarbazone have been studied and the following procedure is proposed. To 1 ml of the soln. (containing ≃25 μg of Mo^{6+}) is added 1 ml of 0·1 *N* H_2SO_4, and after 1 min. the mixture is extracted for 30 sec. with 10 ml of a soln. (0·0025 *M*) of diphenylcarbazone in *iso*amyl acetate. After 5 min. the organic phase is separated, then centrifuged for 2 min., and the extinction at 540 mμ is measured. A blank is carried out with the reagent and the concn. of Mo^{6+} is derived from a calibration graph. If V is present, the pH is lowered by using more concentrated (0·5 *N*) H_2SO_4. The use of the method for special steels is described; modifications are necessary if W is present. L. A. O'NEILL

4750. **Volumetric determination of tungsten in its minerals.** L. Perrier (Comp. Ind. des Metaux Electroniques, Saint Pierre-de-Rumilly, Haute-Savoie, France). *Bull. Soc. Chim. France*, 1959, **26** (1), 177.—*Procedure*—The mineral (2·5 to 3·0 g) is ground, sieved and roasted and fused for 1 hr. with 12 g of Na_2CO_3 - K_2CO_3, and dissolved in < 150 ml of water. Manganese is oxidised by boiling with H_2O_2. The soln. is filtered into a 250-ml flask, and the filtrate and washings are made up to vol. A 50-ml aliquot is neutralised to methyl orange with aq. H_2SO_4 (1:2), and 5 ml of H_3PO_4 (*d* 1·71) and 10 ml of aq. H_2SO_4 (1:2) are added. The soln. and rinsings are poured into a Jones reductor prepared according to Charlot and Bezier (*"Analyse Quantitative Minérale,"* Masson, 1955, 3rd Ed., p. 590), and after exactly 30 min. are run into 40 ml of 10% $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln. and 4 ml of H_3PO_4 (*d* 1·71). The column is washed

out into the flask and the contents are titrated with 0·1 *N* $KMnO_4$. Molybdenum and V, if present, interfere. E. J. H. Birch

4751. **Determination of the solubility of barium tungstate by means of the radioactive isotope tungsten-185.** I. E. Zimakov (State Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1959, **25** (2), 133–134.—The solubility (in mg of W per 100 ml) of $BaWO_4$ in water is 0·729 at 0°, 0·650 at 4°, 0·641 at 10°, and 1·522 at 99°. The solubility *vs.* temp. curve is almost linear from 10° to 99°. The effect of changes of pH (by the use of HCl and NaOH) is shown graphically. The min. solubility is at pH 6 to 7 and this is accordingly the best pH for the separation of W and Re. G. S. Smith

4752. **Application of ion exchange to the colorimetric determination of trace amounts of uranium using dibenzoylmethane.** H. J. Seim, R. J. Morris and R. G. Pastorino (Univ. of Nevada, Reno, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 957–958.—After purification on anion exchangers (*cf.* Seim *et al.*, *Anal. Abstr.*, 1957, **4**, 2969), U is complexed with dibenzoylmethane (**I**) in 80% ethanol and determined colorimetrically at 400 mμ. The method is suitable for the routine determination of U in ores, interference by Th and $Ca_3(PO_4)_3$ being minimised by a double column-adsorption. For U contents of 0·01 to 0·1%, the average deviation is ⩾ 0·002%. *Procedure*—Completely free the eluate from the anion exchanger, containing the U, from $HClO_4$ by evaporation and heating at 250° to 300° for 10 to 20 min. Cool, add water (10 ml) and 95% ethanol (25 ml), and a 1% ethanolic soln. of **I** (1 ml) Bring to pH 7·0 to 7·5 with 0·005 *M* NaOH or H_2SO_4, dilute to 50 ml with 95% ethanol, filter, and measure the extinction at 400 mμ against a blank (1 ml of 1% **I** soln. in 50 ml of 80% ethanol). J. P. Stern

4753. **Machine for preparing phosphors for the fluorimetric determination of uranium.** R. E. Stevens, W. H. Wood, K. G. Goetz and C. A. Horr (U.S. Geol. Survey, Denver, Colo., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 962–964.—The machine described is designed for use in the procedures of Grimaldi *et al.* (*U.S. Geol. Survey, Circ.* No. 199, 1952). Phosphors prepared during the same run show good reproducibility, and separate runs show good agreement. The operation of the machine requires little skill and phosphors can be prepared three times as rapidly as by hand. K. A. Proctor

4754. **Accurate determination of uranium in the presence of small amounts of molybdenum. Lead reductor and stannous chloride methods.** J. T. Byrne, M. K. Larsen and J. L. Pflug (Rocky Flats Plant, Dow Chemical Co., Denver, Colo., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 942–945.—Low and erratic results are obtained when metallic uranium containing 100 p.p.m. of Mo is analysed by the lead-reductor method after fuming the sample with $HClO_4$, but when the sample is fumed with H_2SO_4, Mo (2000 p.p.m.), Fe (1000 p.p.m.) and Si (200 p.p.m.) do not interfere. The interference is attributed to the Mo-catalysed reduction of ClO_4^- by lead, giving a product which oxidises U^{4+}. A comparison of the lead-reductor method (fuming with H_2SO_4) with the Jones reductor method shows the latter to be unsatisfactory for Mo concn. > 50 p.p.m. Analyses by the $SnCl_2$ method are accurate for concn. of Mo ⩾ 6·6% if Mo is determined separately and a correction applied on the basis of a three-electron change for the Mo. is no interference from Al, Ca, Ni and Zn (2 the $SnCl_2$ method, but Cr and Zr cause low and Cu (2%), Ti, W and V high ones. J. P. St

4755. **Spectrophotometric determination of gram quantities of molybdenum in uranium t using dithiol.** A. W. Ashbrook (Res. and Div., Eldorado Mining & Refining, Ltd., O Ontario, Canada). *Chemist Analyst*, 1959, 5–7.—Dissolve a portion of UO_3 containing 8 μg of Mo in 100 ml of HCl (1:1) with he Transfer to a separating-funnel, cool to 30° t Add 0·2 to 0·3 g of hydrazine sulphate, and to dissolve. Add 2 ml of dithiol soln. (0·2% NaOH soln.), mix, and allow to stand for 1 Add 10 ml of CCl_4, shake vigorously for 1 and allow to separate. Filter the CCl_4 layer th a dry paper and measure its extinction in a cell at 670 mμ against a CCl_4 blank. Refe results to a calibration curve. Beer's law is o up to 10 μg of Mo. Results for amounts from 0·1 to 0·26 p.p.m. in high-purity UO_3 good replication. R. E. Ess

4756. **Radiometric analysis of leached uran thorium ore samples with the β-γ-γ method.** de Lange (National Physical Res. Lab., Pre S. Africa). *Anal. Chem.*, 1959, **31** (5), 812–8 In this procedure, the ordinary β-γ method ca tions are made after the ThO_2 concn. has determined, using the 2·6-MeV γ-photopeak ^{208}Tl. The results obtained by this method well with chemical determinations of U_3O_8 and The minimum concn. which can be determi 0·1% in an 18-g sample of ore. K. A. Proc

4757. **Quantitative determination of halog a single drop.** H. Ballczo and L. Hainl (Chem. Inst., Univ., Vienna). *Mikrochim.* 1959, (3), 466–472.—Fractional pptn. of I^- and Cl^- (0·05 to 1 micro-equiv. of halide) diss in a single drop (0·001 to 0·05 ml) is carried o placing the drop in the centre of a White R filter-paper impregnated with Ag oxalate. suitable development by chemical or p chemical means, the annular zones may be r differentiated. The areas of the zones are mea and the ratio of these areas to the area c surface impregnated with Ag oxalate form basis of the procedure for determining the ab amounts of halides in the test drop. The re accuracy is ±10%. A similar procedure m used for ensuring the uniform distributi carrier-free radioactive iodine over a paper which corresponds to that of the counting thus reducing the error of the counting tech D. F. Phil

4758. **Micro-determination of fluorine appl to atmospheres and to biological media.** R. R. Truhaut and J. Bernuchon (Lab. Toxic d'Hygiène Ind., Fac. Pharm., Paris). *C Rend.*, 1959, **248** (4), 504–507.—After collecti the sample (dust filtered from air, or va aspirated through dil. NaOH soln.), the F separated from interfering ions (PO_4^{3-}, SO_4^{2} distillation in superheated steam in the prese H_2SO_4 and of SiO_2 (Fabre *et al.*, *Ibid.*, 1955 226). From 1 to 100 μg of F^- in the neutr distillate can then be determined by titration 0·004 *M* $Th(NO_3)_4$ in a white porcelain dish the colour of the indicator {1 ml of 0·02% so

NS [sodium 1:8-dihydroxy-2-(4-sulpho-lazo)naphthalene-3:6-disulphonate] plus 1 ml I 3·1 buffer} changes from red to violet. ccuracy is within 3%, and the sensitivity is d to be adequate for the control of industrial ıe. The procedure is applicable to biological es (H_2O, alcoholic drinks, bone, blood and provided that F^- are fixed by ignition at with a mixture of MgO and Mg acetate and en liberated by distillation.

W. J. Baker

). New spot test for the detection of fluoride R. Belcher, M. A. Leonard and T. S. West . Dept., Univ., Edgbaston, Birmingham, nd). *Talanta*, 1959, **2** (1), 92–93.—Fluoride hange the red colour of the Ce^{III} chelate of n complexone (1:2-dihydroxyanthraquinon-ethylamine-NN-diacetic acid (**I**) to lilac blue. *lure*—Mix one drop of the test soln. with one of a 0·001 *M* soln. of **I** buffered to pH 4·3 Na acetate - acetic acid. Add, with stirring, rop of 0·001 *M* $Ce(NO_3)_3$. The limit of identi-n is 0·2 μg and the dilution limit 1 in 1·8 × Serious interference is caused by phosphate, ; organic chelating agents and certain heavy s.

W. T. Carter

0. Amperometric determination of fluoride rotated aluminium electrode. I. M. Kolthoff C. J. Sambucetti (Sch. of Chem., Univ. of sota, Minneapolis, U.S.A.). *J. Amer. Chem.* 1959, **81**, 1516–1517.—A rotated aluminium ode ($\frac{1}{16}$ in. diam., 0·15 sq. cm surface area, .p.m.) is readily polarised in HNO_3 soln. of 2, and in a soln. buffered with acetate at to 5·5. In the absence of O, the limiting nts at − 0·75 V are proportional to the F^- . from 10^{-5} *M* to 3×10^{-4} *M*. Amperometric ions with $Al(NO_3)_3$ soln. in an acetate basal at pH 3·5 give good results. It is claimed the electrode acts as a pF electrode in acid The results of Baker and Morrison (*cf. Anal.* , 1955, **2**, 3350) could not be reproduced.

T. R. Andrew

1. Determination of small amounts of fluorine ods and water. II. Qualitative detection of le. K.-E. Quentin, J. Indinger and S. W. (Balneologische Inst., Univ. Munich, any). *Z. LebensmittUntersuch.*, 1959, **109** 13–218.—Fluorine is isolated as HF from aq. (concentrated by evaporation if necessary) by -distillation into a suspended drop of water Feigl apparatus, which consists of a weighing , the stopper of which has a projection on nderside for the suspension of the drop of . The test soln. (a few ml) is gently heated a few drops of H_2SO_4 and a little tartaric acid e point of incipient browning, after which rop of water (containing 25 to 33% of the ble HF) is transferred to a spot-testing tile, ested in comparison with standard soln. of (5 to 250 μg per litre) by the addition of 50% acid and zirconium - haematoxylin indicator drop of each). As little as 0·2 μg of F can be ted by the colour change. For a semi-quant. nination, a factor of 3·5 is applied to com-te for the incomplete recovery of the available om the test soln.

P. S. Arup

2. Determination of small amounts of fluorine mina-containing materials. E. Abrahamczik W. Merz (Badischen Anilin- & Soda-Fabrik A.-G., Ludwigshafen/Rhein, Germany). *Mikrochim. Acta*, 1959, (3), 445–455.—Fluorine in ores and catalysts containing Al_2O_3 as carrier material may be rapidly determined down to 5 p.p.m. with a relative accuracy, depending on the F^- content, of between 2 and 5%. The separation of F^- (as HF) is cleanly and selectively accomplished from most other interfering elements by fusing the specimen (1 g) with $K_2S_2O_7$ in a quartz flask having a side-arm which projects into the melt and through which a strong air-blast is led to drive off the HF, which is absorbed in *N* NaOH. Quantitative removal of the remaining traces of interfering ions is carried out by steam-distillation from $HClO_4$ into 0·1 *N* NaOH. The determination is finished by titration with $Th(NO_3)_4$ soln., with a mixed indicator of Na alizarinsulphonate and methylene blue.

D. F. Phillips

4763. **Argentimetric determination of chloride by Volhard's method and a study of various modifications.** O. Alcides Ohlweiler, J. de Oliveira Meditsch and S. Kuperstein (Escola de Engenharia, Porto Alegre, R.G.S., Brazil). *Chim. Anal.*, 1959, **41** (3), 94–96.—Suggested modifications to Volhard's method to eliminate error due to reaction between AgCl and SCN^- (filtration of AgCl, addition of nitrobenzene, or of large amounts of Fe^{3+}) are critically examined. Three procedures are recommended, the first being the most accurate. (*i*)—Weigh 10 g of 0·4 *N* NaCl (from a weight burette) into a beaker, add H_2O (10 ml) and 6 *N* HNO_3 (5 ml) and a weighed excess of 0·1 *N* $AgNO_3$ (2 to 5 g excess). Stir for 5 min. then filter and wash the ppt. with HNO_3 (1:99) (10 × 10 ml). To the filtrate and washings add 40% $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln. (1 ml) and a weighed amount of 0·1 *N* KSCN, then complete the titration with 0·01 *N* KSCN to a red colour stable for 1 min. (*ii*)—To 10 g of 0·4 *N* NaCl add H_2O, HNO_3 and $AgNO_3$ soln. as before. Add nitrobenzene (3 ml), then $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln. and 0·1 *N* KSCN, and complete the titration as before. (*iii*)—To 10 g of 0·4 *N* NaCl add H_2O (10 ml) and 6 *N* HNO_3 (17 ml), then 2 *M* $Fe(NO_3)_3$ (10 ml) and 1·00 ml of 0·01 *N* KSCN. Add a weighed amount of 0·1 *N* $AgNO_3$ and complete the titration with 0·01 *N* $AgNO_3$ to the discharge of the red colour. Standard deviations for the 3 methods are ±0·00024, ±0·00041 and ±0·00030 g, respectively (each based on 5 determinations).

P. D. Parr-Richard

4764. **Precision null-point potentiometry. A simple, rapid and accurate method for determination of low concentrations of chloride.** H. V. Malmstadt and J. D. Winefordner (Univ., Urbana, Ill., U.S.A.). *Anal. Chim. Acta*, 1959, **20** (3), 283–291.—With the apparatus and procedure described, concn. of Cl^- from 10^{-6} to 10^{-1} *M* can be determined in a few minutes with an error of $\simeq$ 0·1%. The concn. of Cl^- in the sample is changed, while keeping the ionic strength constant, until it is identical with that of the standard reference soln., *i.e.*, zero potential is attained across the two silver - silver chloride electrodes, one in each soln. If the concn. of Cl^- in the sample soln. is higher than that in the reference soln., dilution to the null point is effected by the addition of *M* H_2SO_4, but if it is lower a soln. twice as conc. in Cl^- as the reference soln. is added. The ionic strength of all soln. is kept constant by the addition of *M* H_2SO_4 and diluting to the correct vol. The only calculation needed is either a simple addition or subtraction; the control of temp. and corrections for

Cl^- in the reagents, etc., are obviated. The method is specially applicable when the concn. of Cl^- in a series of samples varies over a relatively small range. There is a slow drift of electrode potential when Br^- are present. W. J. Baker

4765. **Rapid determination of chloride in silica or other solids.** J. W. Robinson (Esso Standard Oil Co., Baton Rouge, La., U.S.A.). *Anal. Chim. Acta*, 1959, **20** (3), 256–258.—The sample (> 1·5 g) is heated to fuming in a porcelain boat with a mixture (9:1) of conc. H_2SO_4 and conc. HNO_3 in a furnace (max. ≃ 590° F) in a stream of N. The evolved Cl^- are collected in 100 ml of 2% Na_2CO_3 soln. and determined either colorimetrically with $Hg(SCN)_2$ and $(NH_4)_2SO_4.Fe_2(SO_4)_3$, or by potentiometric titration in acetone soln. with $AgNO_3$ soln. at pH 2. An analysis takes ≃ 20 min. and the accuracy is satisfactory. W. J. Baker

4766. **Determination of chlorides in zinc electrolytes by non-compensated potentiometric titration.** Z. I. Ivanova and P. N. Kovalenko (Rostov State Univ.). *Zavod. Lab.*, 1959, **25** (3), 290–291.—With a mercury indicator electrode and a tungsten reference electrode the soln. is titrated with 0·1 *N* $Hg(NO_3)_2$. G. S. Smith

4767. **Simultaneous determination of chlorite and hydrogen peroxide in the presence of chlorides, chlorates, carbonates and caustic alkalis [in production control of sodium chlorite].** A. I. Kachalov and I. G. Bŷkova. *Zavod. Lab.*, 1959, **25** (3), 289–290.—The sample (10 ml) is boiled with 100 ml of water and 0·2 to 0·4 g of PbO_2 for 10 to 15 min., the cooled soln. is treated with 20 ml of 10% KI soln. and 10 ml of dil. H_2SO_4 (1:1), and the iodine liberated is titrated with 0·1 *N* $Na_2S_2O_3$ to give the $NaClO_2$ content. The total of H_2O_2 and $NaClO_2$ is determined on another portion without the use of PbO_2, and H_2O_2 is obtained by difference. G. S. Smith

4768. **Coulometric titrations of mixtures of perchloric acid and acetic acid in water - acetone medium.** R. Rosset and B. Trémillon (Lab. de Chim. Anal., École Sup. de Phys. et de Chim. Ind., 10, rue Vauquelin, Paris, France). *Bull. Soc. Chim. France*, 1959, **26** (1), 139–144.—In acetone - water soln. (4:1), $HClO_4$ and acetic acid are neutralised independently at different potentials. An arrangement is described in which the soln. is electrolysed at constant current, with $NaClO_4$ as basal electrolyte, with a platinum cathode providing the OH^- and a silver - silver chloride electrode dipping into NaCl soln. separated from the cathode compartment by a sintered glass disc. The indication of the two end-points is effected by plotting the current in an indicator electrode circuit with ≃100 mV constant p.d. or with 10 to 15 mV constant p.d. Alternatively, the voltage of the indicator electrodes for a constant current is measured. In this way mixtures ≃0·001 *M* with respect to each acid are determined to within ≃1%. The theory of the coulometric neutralisation and of the function of the indicator electrodes is discussed. E. J. H. Birch

4769. **Titration of periodates in the presence of iodate with hydrazine sulphate.** A. Berka and J. Zýka (Dept. Anal. Chem., Charles' Univ., Prague). *Českosl. Farm.*, 1959, **8** (3), 136–138.—In soln. buffered with $NaHCO_3$, borate or Na acetate (pH 7·5 to 9), periodate can be titrated potentiometrically with a soln. of hydrazine sulphate accord the equation—

$$2IO_4^- + N_2H_4 = 2IO_3^- + N_2 + 2H$$

The determination of 10 to 20 mg of KIO_4 carried out in the presence of a 20-fold ex iodate. The procedure is reliable for the ti of the excess of periodate in the indirect mination of organic compounds. J. Z

4770. **Titrimetric determination of man following nitric acid oxidation in the prese pyrophosphate.** C. O. Ingamells (Rock Anal Univ. of Minnesota, Minneapolis, U.S.A.). *T* 1959, **2** (2), 171–175.—Bivalent Mn is oxidi Mn^{III} and titrated with ferrous soln., wi phenylamine as indicator. To the Mn preferably containing HNO_3 as the only aci 10 ml of 85% H_3PO_4 plus an additional 15 each 40 mg of MnO expected. Add 5 ml of and evaporate on a hot-plate, without boili the vol. is reduced to that of the H_3PO_4 Then add 5 to 6 g of $Na_4P_2O_7$ for each 20 H_3PO_4 and heat till the HNO_3 is expelled, as by absence of condensation on the walls flask. Add 1 ml of HNO_3 (1:1, boiled to r oxides of N) and heat for 10 to 15 min., but out removal of all HNO_3. Cool until the fla be handled, then add one-quarter of the cold water, mix, and cool in running Dilute with water to about 20% in H_3PO in running water, and titrate with 0·1 or $(NH_4)_2SO_4.FeSO_4$ soln. in 5% H_2SO_4 (freshl pared with boiled water), adding the ind (1% soln. of diphenylamine in conc. H_2S 0·2% aq. Ba diphenylaminesulphonate soln. before the end-point, when the pink colo Mn^{III} is almost discharged. The end-point i sharp. With > 40 mg of MnO in 15 ml of oxidation may be incomplete, a safe limit 2 mg of MnO per ml. Results for garnet, phos minerals, bauxite, Ni - Cr steel, and Fe an ores agree well with those by gravimetri colorimetric procedures. Modifications re for the preliminary dissolution of some of materials are discussed. R. E. Ess

4771. **Micro-determination of iron by mea catalysis.** L. Erdey and F. Szabadváry (I allg. Chem. der Tech. Univ. Budapest, Hun *Mikrochim. Acta*, 1959, (3), 424–431.—The re between H_2O_2 and Variamine blue is catalys Fe^{III}, and this effect can be utilised for the mination of Fe. The method is based on me ment of the time required for the photo galvanometer to reach a pre-determined division for a sample to which no Fe has added. This measurement is then repeated presence of a known amount of Fe. Final reaction time is measured on the sample on Fe is to be determined. The unknown amo Fe may be calculated from the three reaction By this means it is possible to determine as as 2 × 10^{-9} g of Fe per ml. D. F. Phil

4772. **Analytical applications of the comple XVI. Indicators for the macro- and volumetric determination of iron with 1:2-dia *cyclo*hexane-NNN′N′-tetra-acetic acid.** F. Be Martínez and R. Rey Mendoza (Lab. de Quím. Fac. de Ciencias, Santiago de Compostela, S *Quím. e Ind., Bilbao*, 1959, **6** (1), 3–7.—S salicylic acid (**I**), tiron (**II**) and KSCN were as indicators for the volumetric determinat

with 1:2-diamino*cyclo*hexane-NNN′N′-tetra- acid. The best results were given by **I** of 2% soln.) at 50° and pH 2 to 5, but they l to 3% high; the best results for **II** (2 ml of ɔln.) at 50° and pH 2 to 4 were 1 to 4% high. r results were obtained when KSCN (2 *M*) ısed, although the amount of indicator had effect on the results. For soln. containing 120 mg of Fe, and 15 ml of KSCN soln., ɛsults were up to 0·5% high. It was found ɜary to use 10 ml of amyl alcohol or diethyl to obtain a sharp end-point in the micro- ɪon of soln. containing up to 12 mg of Fe, results were then within ± 0·5% of the ɛtical. G. H. Foxley

3. **Photometric titration of iron with ethylene- nedi-(o-hydroxyphenylacetic acid).** A. L. rwood (Emory Univ., Atlanta, Ga., U.S.A.). *Chim. Acta*, 1959, **20** (3), 228–232.—Less l mg of Fe^{3+} in 100 ml of soln. can be titrated 3·5 with a 0·01 *M* soln. of ethylenediamine- ıydroxyphenylacetic acid), the end-point found photometrically at 470 mμ. The error 1%; Th, Ag, Cu and Al interfere, but not Ca, Cr^{III}, Mn^{II}, Co^{II}, Zn, Cd, Hg^{II}, Pb and Microgram amounts of Fe can be determined aling down the vol. of soln. and the titrant ., and milligram amounts by changing to a ength (550 mμ) at which the sensitivity is W. J. Baker

4. **Spectrophotometric determination of iron 3-hydroxy-3-methyl-1-phenyltriazen.** H. K. L. a and N. C. Sogani (Chem. Dept., Gov. Coll., r). *J. Indian Chem. Soc.*, 1959, **36** (2), 87– Ferric iron forms a greenish blue, water- le complex with 3-hydroxy-3-methyl-1-phenyl- n $[C_6H_5N:N{\cdot}N(CH_3)OH]$ in the pH range ɔ 4·5, with an absorption peak at 625 mμ. reaction is used for the direct spectrophoto- c determination of Fe in the presence of almost e commonly associated elements. The system Beer's law in the concn. range 1 to 10 p.p.m. e. The sensitivity of the colour reaction mined in Nessler cylinders is 1 part of Fe in 10^6 parts of soln. and on a spot plate is g of Fe. The complex has the formula FeR_3 reagent). The molecular extinction coefficient 5 mμ and the dissociation constant at 34° ·8 × 10^3 and 1·156 × 10^{-11}, respectively. I. Jones

5. **Determination of bivalent and tervalent with a dithiocarbamoylhydrazinium salt by metric or spectrophotometric means.** A. Musil V. Haas (Inst. f. anorg. u. anal. Chem., Univ., Austria). *Mikrochim. Acta*, 1959, (3), 481– –Bivalent Fe reacts with a dithiocarbamoyl- zinium salt to form a violet complex in acetate soln., which has an absorption max. at 570 This reaction may be used for the determina- f Fe^{2+} in the concn. range 0·01 to 5 μg per ml. reduction with ascorbic acid, Fe^{3+} may also etermined by this method. The interfering s of other cations and their max. permissible . have been determined. D. F. Phillips

6. **Rapid method of determining iron in nt.** N. S. Frumina (N. G. Chernyshevskii ov State Univ.). *Zavod. Lab.*, 1959, **25** (2), 49.—The sample (0·25 g) is dissolved by g with 10 ml of conc. HCl containing 0·5 g H_4Cl, and the soln., without being filtered, is ed hot with 0·5 *N* $SnCl_2$ until the colour is light yellow. Indigo carmine soln. (25%) (3 to 4 drops) is added and the titration is continued slowly until the colour changes from green to blue and then to colourless. Addition of 0·05 *N* $K_2Cr_2O_7$ (possibly 3 or 4 drops) is now made until a stable blue colour appears. (If one drop is sufficient just the right amount of $SnCl_2$ soln. has been used.) The excess of $K_2Cr_2O_7$ is determined by titration with a soln. of Fe^{2+} in the presence of 3 to 4 drops of 0·1% phenylanthranilic acid as indicator. The method takes 10 to 12 min. G. S. Smith

4777. **Gases in ferro-alloys.** N. Christensen (SINTEF, Tech. Univ. of Norway, Trondheim). *Tidsskr. Kjemi Bergv.*, 1959, **19** (3), 57–61.—The chemical bonding of O, N and H in different ferro-alloys is discussed. Typical analytical data on the contents of O, N and H in some commercial alloys are presented. The estimated amount of gaseous impurities introduced into steel baths by ferro-alloy additions is given. The sampling of ferro-silicon and the determination of H in that alloy is discussed (Christensen and Gjermundsen, *J. Iron St. Inst.*, 1958, **190**, 248). B. Rönnholm

4778. **Apparatus for carbon content [of steel] determination.** R. Rew (B.C.I.R.A. Res. Dept., Bordesley Hall, Alvechurch, Birmingham). *J. Res. Brit. Cast Iron Ass.*, 1959, **7**, 651–655.—An improved layout, furnaces and oxygen control for the determination of C in iron and steel are described. C. H. Cowper-Coles

4779. **Determination of major constituents in alloy steels by spectrographic solution methods.** C. A. Waggoner (Pacific Naval Lab., Esquimalt, B.C., Canada). *Appl. Spectroscopy*, 1959, **13** (2), 31–33.—Stainless steels can be analysed spectrographically by adapting either the porous cup or rotating disc soln. techniques. The procedure described is most useful if no standard alloys are available and if the samples vary in size and shape. Cobalt is used as an internal standard and Fe, Cr, Ni, Mn and Mo can be determined to within ± 3% of the amounts present. Inter-element effects arising from wide variations in composition are negligible. The method could probably be extended to low-alloy steels and nickel-base alloys containing Fe and Cr. K. A. Proctor

4780. **Alkalimetric determination of phosphorus [in steel] as quinoline molybdophosphate.** G. Alfelt (Air Force Central Lab., Malmslätt, Sweden). *Jernkontor. Ann.*, 1959, **143** (3), 167–174.—The pptn. of quinoline molybdophosphate (**I**) in various media was studied. In 0·5 *M* to 5 *M* HNO_3 the pptn. of **I** is quant. The presence of Fe^{3+} as nitrate alters the highest permissible acid concn. to 4 *M* HNO_3 and as chloride to 3 *M* HNO_3. Tartaric acid (< 0·4 g) has no influence on the pptn. In 0·5 *M* to 1·75 *M* HCl the pptn. of **I** is quant. In the presence of Fe^{3+} the highest permissible acid concn. is 1 *M* HCl. In 1 *M* to 4 *M* $HClO_4$ the pptn. of **I** is quant. Generally the amount of reagent required for the quant. pptn. of **I** is increased in more acid soln. *Reagent*—Dissolve $Na_2MoO_4.2H_2O$ (100 g) in H_2O (250 ml), add HCl (sp. gr. 1·19, 115 ml) and HNO_3 (sp. gr. 1·40, 100 ml). Add this soln. to a soln. of quinoline (14 ml) in a mixture of H_2O (160 ml), conc. HCl (75 ml) and conc. HNO_3 (60 ml). Add H_2O (500 ml) and filter after 24 hr. Store in a polyethylene vessel. *Procedure for steel soluble in HNO_3*—Dissolve the sample (2 g) in HNO_3 (1:1) (40 ml), heat to

boiling, add $KMnO_4$ soln. (3%, w/v) (5 ml), boil, add tartaric acid soln. (20%, w/v) (2 ml), boil, add H_2O (80 ml), heat to boiling and add reagent (25 ml). Filter, wash the ppt. with 1 *M* HNO_3 and 0·2 *M* KNO_3, then dissolve it in H_2O and standard NaOH soln. Back-titrate with standard HCl soln. against a mixture (2:3) of phenolphthalein and thymol blue (both 0·1% in ethanol). Procedures are also given for steel insoluble in HNO_3. The reported results show good precision and accuracy. B. Rönnholm

4781. **Colorimetric determination of traces of selenium in steel. I. Determination of small quantities of selenium in the absence of other elements.** N. Leontovitch (Inst. Rech. de la Sidérurgie, Saint-Germain-en-Laye, France). *Chim. Anal.*, 1959, **41** (2), 56–62.—In the preliminary investigation described, the effect of reagents and conditions on the production of suspensions of Se by reduction was studied. Reduction with hydrazine hydrochloride in the presence of formic acid has certain advantages, *viz*, in moderate concn. formic acid is a good solvent for tellurium dioxide, it does not absorb significantly below 270 mμ, neither tellurous nor telluric acid (in amounts of 1 mg of Te per 50 ml) increases the extinction of 6 *N* formic acid, and oxygenated compounds of Te are not reduced under the conditions used. *Procedure*—The test soln. (10 ml) (containing > 500 μg of Se in the form of an oxygenated compound) is placed in a 25-ml flask and the following reagents are added successively, with shaking after each addition—10 ml of 15 *N* formic acid, 2·5 ml of poly(vinyl alcohol) soln. (20 mg per ml), 1 ml of 10% hydrazine hydrochloride soln. and water to 25 ml. A reference soln. is made from the same reagents and subjected to the same treatment. After being heated at 93° to 95° in a boiling-water bath for 5 min. the soln. is cooled and the extinction is measured at 315 mμ in a 1-cm cell and compared with that of the blank. Standards should be prepared for each series of determinations and used to give absolute values. The method is sensitive to 2 μg of Se per ml. S. M. Marsh

4782. **Determination of the tungsten content in alloyed steels by the β,γ-reflection method.** J. Fodor (Csepel Iron and Metal Works, Budapest). *Acta Chim. Acad. Sci. Hung.*, 1959, **19** (4), 327–335 (in English).—By measurement of the radiation from a surface irradiated by a β-source, W in steel can be determined within 1% absolute. The source is a film of ^{204}Tl deposited on the inner surface, inclined at an angle of 45°, of a copper ring. The radiation returning through the ring consists of reflected β- and secondary γ-radiation, which is measured with a scintillation counter. One type of radiation characteristic of W is selected by a discriminator. The radiation to be selected is established with the aid of a tungsten plate. The calibration curve is rectilinear from 1% to 18% of W. Sampling takes 2 min. and the measurement takes 100 sec. The activation method used to check the calibration samples is described. G. Burger

4783. **Sodium *p*-(mercaptoacetamido)benzenesulphonate as colorimetric reagent for cobalt.** H. K. L. Gupta and N. C. Sogani (Gov. College, Ajmer, India). *Anal. Chem.*, 1959, **31** (5), 918–920.—Sodium *p*-(mercaptoacetamido)benzenesulphonate (**I**) forms water-sol. brownish-red complexes (Co - $\mathbf{I}_3$) with Co^{II}. Between pH 6·5 and 7·5 the absorption max. is at 475 m between pH 8·5 and 11·0 at 390 mμ. Both are suitable for colorimetry but that at is preferable; Ni^{II} and Fe also form com and interference by Ni cannot be com excluded except by separating the metals. law holds in the range 0·3 to 2 p.p.m. of Co ducibility not given). The method is suita the determination of 15 to 100 μg of Co presence of Zn (200), Mn (200), Cu (150), Fe Al (300), Cr (150), Ag (150), Pd (5), WO_4 and MoO_4^- (150 p.p.m.). *Procedure*—To soln. (15 to 100 μg of Co) add 10% Na K ta soln. (2 ml), buffer (pH 7·0) (5 ml) and 1% (5 ml). Place in a boiling-water bath for min., make up to 50 ml, and read the ext at 475 mμ against H_2O. In the presence adjust the pH to between 2 and 4 with HCl dilution. J. P. St

4784. **Determination of trace amounts of in iron and steel and some associated ma** BISRA Methods of Analysis Committee. *J St. Inst.*, 1959, **191** (3), 236–240.—The mended method is satisfactory for deter < 0·04% of Co in iron and steel, ferro-all Mn, Ti, Ni and Cr, chromium and nickel meta iron ore. Reproducibilities of ±0·001 > 0·01% of Co and ±0·002% for > 0·04% of claimed. The method involves photo measurement of the cobalt complex with n R salt after separation by ZnO and pptn. with 1-nitroso-2-naphthol. J. W. O. Pyem

4785. **New colour reaction for the identif of the nickel ion.** S. Popa and G. Furnică. *R.P.R., Stud. Cercet. Chim.*, 1959, **6** (3), 481– The study of the microcrystalline structure sodium, ammonium and nickel salts of ethy phono- and diethylphosphino-dithioic acids e these two acids to be separated and ider The nickel salt of the former acid gives a cha istic violet-coloured ether extract; Zn^{2+}, Mn^{2+}, Ba^{2+}, Mg^{2+}, Cd^{2+}, Cu^{2+}, Ca^{2+}, Bi^{3+}, Al^{3+}, Ag^+, Pb^{2+} and K^+ do not interfere. The y green colour given by the ether extract Co^{2+} salt masks that of Ni^{2+} only if the C large excess, but this in turn can be supp by a large excess of the organic reagent. sensitivity of the reagent is only slightly les that of dimethylglyoxime (≃ 1·31 μg), but more specific. H. S

4786. **Spectrophotometric determination of of nickel with 4-*iso*propyl*cyclo*hexane-1:2 dioxime.** B. L. McDowell, A. S. Meyer, R. E. Feathers, jun., and J. C. White (Oak Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 19 (5), 931–934.—The high extraction coeff. complex of Ni^{2+} with 4-*iso*propyl*cyclo*hexan dione dioxime (**I**) in xylene permits the extr of 10 μg of Ni from 500 ml of aq. soln. a spectrophotometric determination with an ac within ± 2%. Co-extracted Cu (> 800 μ be removed from the xylene phase by a singl with 5% thioacetamide soln. (5 ml). Interf by Fe (200-fold excess) can be masked (0·6 g). *Procedure*—Adjust the pH of the aq of Ni (10 to 150 μg) to approx. 7 and add su 10 *M* ammonium acetate to make the fina 1 *M* with respect to acetate. Add 2 ml of 0· aq. **I** per 100 ml of soln. After 30 min. e with xylene (10 ml) and measure the extinct the xylene soln. at 383 mμ. J. P. St

. **Spectrophotometric determination of nickel ıercaptoacetic acid.** P. C. Christopher and . Hamme (Chemstrand Corp., Pensacola, U.S.A.). *Appl. Spectroscopy*, 1959, **13** (1), –The method is sensitive to 0·001 mg of Ni and Beer's law is followed up to concn. of per ml. The optimum working range, which the error is 3%, is 0·04 to 0·5 mg of ml. The colour produced is stable for hours and is unaffected by changes in pH g as the soln. is alkaline. A preliminary tion of the Ni by dimethylglyoxime extrac- iminates interfering metals, and the addition citrate before development of the colour ts interference from metals extracted with K. A. PROCTOR

. **Precipitation of nickel(II) sulphide from acid ns by thioacetamide.** D. F. Bowersox, D. M. and E. H. Swift (California Inst. of Technol., ena, U.S.A.). *Talanta*, 1959, **2** (2), 142–153.— uation is given for calculating the rate of of Ni^{2+} as sulphide by thioacetamide (**I**) ıcid soln. at 90° ± 1°, and the conditions e separation of Pb and Cd from Ni at 90° H 1 to 2 are discussed. Hydrogen ion concn. me are critical factors in the separation of d and other H_2S-group metals from Ni by with H_2S from soln. of **I**. The equation s the calculation of the amount of Ni which be co-pptd. by direct reaction with **I** under conditions, and it appears that the pptn. Cd, etc., with sulphide from soln. of **I** should rried out at the lowest permissible pH and shortest permissible time to obtain effective tion, and that the ppt. should be removed the soln. as soon as quant. separation has achieved. Precipitation conforming to the on is obtained at H^+ concn. at which pptn. S does not occur, and the rate of formation S is independent of the quantity of metal de present, and of the concn. of Pb^{2+}, Cd^{2+} $_2$S in the soln. It is pointed out that complex ffects may seriously affect equilibrium and onditions, and that caution is needed when tuting **I** for H_2S in analytical separations. R. E. ESSERY

9. **Determination of nickel in gold - nickel** D. H. Wilkins and L. E. Hibbs (Gen. ic Co., Schenectady, N.Y., U.S.A.). *Anal. Acta*, 1959, **20** (3), 273–274.—The Ni is ted by passing the sample soln. (adjusted to HCl) through a long column of strongly basic exchange resin, whereby the Au complex is retained at the top of the column. The Ni ted with 2 *N* HCl, and can then be deter- by the back-titration of an excess of 0·03 *M* with 0·03 *M* CuSO, with 1-(2-pyridylazo)-2- hol as indicator. The error is ≃ + 0·2%. ssive samples can be analysed until the Au ex is near the base of the column. W. J. BAKER

0. **Spectrophotometric determination of ium with 1-naphthylamine-3:5:7-tri- nic acid.** E. L. Steele and J. H. Yoe (Dept. iem., Univ., Charlottesville, Va., U.S.A.). *Chim. Acta*, 1959, **20** (3), 211–215.—From 14 p.p.m. of Ru can be determined by ring the extinction at 530 mμ of the stable mplex formed at pH 2·5 to 5 (dil. HCl) en 1-naphthylamine-3:5:7-trisulphonic acid 10^{-4} *M* aq. soln.) and Ru^{VI}. The reaction is at 15° and the complex, which obeys Beer's law, has the formula $(\text{reagent})_2RuO_4$. All interfering ions should be removed by first boiling off Os as the tetroxide from conc. HNO_3 soln., followed (after addition of $KMnO_4$) by a second distillation to collect RuO_4 in the reagent (5 or 25 ml). The sensitivity is 0·1 μg per ml. W. J. BAKER

4791. **Gravimetric determination of palladium with 2-*o*-hydroxyphenylbenzoxazole.** R. F. Wilson and L. J. Baye (Dept. of Chem., Texas Southern Univ., Houston, U.S.A.). *Z. anal. Chem.*, 1959, **166** (5), 359–361 (in English).—*Procedure*—Palladium is treated with hot fuming HNO_3. To 1 vol. of soln. containing 2 to 30 mg of Pd are added, with stirring, 2 vol. of ethanol and 1 vol. of a satd. ethanolic soln. of 2-*o*-hydroxyphenylbenzoxazole. The ppt. is filtered off immediately on a medium-porosity sintered glass crucible, washed several times with 50% ethanol, and dried at 110° for 1 hr. Cations that interfere are listed. B. B. BAUMINGER

4792. **Spectrophotometric determination of palladium with phenyl 2-pyridyl ketoxime.** Buddhadev Sen (Coates Chem. Lab., Louisiana State Univ., Baton Rouge). *Anal. Chem.*, 1959, **31** (5), 881–884.—The Pd^{II} chelate of phenyl 2-pyridyl ketoxime (**I**) contains 2 moles of **I** per mole of Pd and has absorption max. at 340 and 410 mμ (ε 50,000 and 30,000, respectively) in $CHCl_3$, which can be used for the determination in 1-cm cells of 1·5 to 8 and 2 to 10 p.p.m. of Pd, respectively. The colour of the chelate is stable; 5 mg of Be, Mg, Sr, Ba, Zn, Cd, Hg^{II}, Al, Pb, Bi^{III}, VO^{2+} Mn^{II}, Fe^{III}, Ru^{VI}, Rh^{III}, Os^{VI}, Ir, Pt and Ag do not interfere with the determination of Pd (100 μg). Interference by Cu^{II}, Fe^{II}, Co^{II} and Ni^{II} is eliminated with EDTA. Only Au^{III} and CN^- interfere seriously. *Procedure*—To the Pd soln. add a 1% ethanolic soln. of **I** (2 ml), dilute to 15 ml, adjust to pH 8·5 to 10 with aq. Na_2CO_3 soln. and extract twice with 10 ml of $CHCl_3$. Separate the organic phase and make it up to 25 ml. Measure the extinction. J. P. STERN

4793. **Application of organoselenium compounds in analytical chemistry. II. The determination of palladium and other metals with N-acetyl-N′-phenylselenourea.** F. Pino Pérez, F. Burriel-Martí and L. Martínez Conejero (Dept. de Quím. Anal. del C.S. de I.C. Madrid y La Laguna, Spain). *Inf. Quim. Anal.*, 1959, **13** (2), 38–51.—*Qualitative tests*—One drop of an alcoholic soln. of N-acetyl-N′-phenylselenourea (0·1 g per 100 ml) is added to two drops of a soln. of a metal (0·1 g per 100 ml). Precipitates or turbidities are formed by Os, Pd, Pt, Au, Hg and Ag, and the effects of dil. HNO_3 (1:1), aq. NH_3 (10%) and KCN soln. (10%) on the ppt. are noted. The sensitivity is 1 μg for Pd (dilution limit $6{\cdot}7 \times 10^{-6}$). *Quantitative use*—Bivalent Pd forms a complex with N-acetyl-N′-phenylselenourea, which is stable at pH values < 3 and > 8·4, and which has max. extinction at 470 mμ; Cu^{2+} interfere. The determination of Ag^+ is similar to that for Pd^{2+}, but the determination of Pt^{4+} is unsatisfactory. The selectivity of the reagent is attributed to the –CSe·NH– group. G. H. FOXLEY

4794. **5-Aminothiazoline-2-thiocarboxyamide used as analytical reagent. II. The palladous compounds, their composition and analytical applications—heterometric titration.** Ping-Shih T'ien and K'uei Wang (Dept. of Chem., Peking Med.

Coll.). *Acta Chim. Sinica*, 1959, **25** (2), 85–91.—5-Aminothiazoline-2-thiocarboxyamide (**I**) (*cf. Anal. Abstr.*, 1958, **5**, 4145) is used in the determination of micro quantities of Pd by heterometric titration in 95% ethanol with an error $\gtrless \pm 3{\cdot}5\%$. EDTA is added to obviate interference by foreign metals, *e.g.*, Ca, Ba, Zn, Mn, Co, Fe, Cr, Pb, Pt, Cu, Ce and Cd. In weakly alkaline aq. and alcoholic soln., two palladous compounds with the ratios of **I** to Pd of 1:1 and 2:1 have been identified. S. H. YUEN

4795. **Rapid and precise micro-heterometric determination of gold, palladium or platinum with nitron in thiocyanate solutions. A study of the reactions and compounds.** M. Bobtelsky and J. Eisenstadter (Hebrew Univ., Jerusalem, Israel). *Anal. Chim. Acta*, 1959, **20** (3), 216–227.—Procedures are given for the determination, in $\simeq 10$ min., of Au^{III} ($\simeq 1$ mg), Pd^{II} ($\gtrless 0{\cdot}5$ mg) and Pt^{IV} ($\simeq 1{\cdot}5$ mg), either alone or in mixtures, by heterometric titration with 0·01 *M* nitron soln. at pH 1 to 7 in the presence of an excess of KSCN. The sample soln. ($\simeq 20$ ml) can contain HCl, HNO_3, H_3PO_4 or acetic acid as well as any of the following masking agents in excess—citrate, tartrate, EDTA (disodium salt), $P_2O_7^{4-}$ and $P_3O_{10}^{5-}$. The error is $<$ 1%. The insol. compounds obtained quant. at the end-points of the titrations are AuN, Au_2N_3, Pd_2N_3 and Pt_4N_3 (N = nitron). When determining Au, the sensitivity is increased if the heterometric titration is made in a soln. containing a large excess of aq. NH_3 ($\simeq 0{\cdot}3$ *M*), the end-point occurring at Au_2N_3. W. J. BAKER

4796. **Spectrophotometric determination of osmium with 1-naphthylamine-4:6:8-trisulphonic acid.** E. L. Steele and J. H. Yoe (Dept. of Chem., Univ., Charlottesville, Va., U.S.A.). *Anal. Chim. Acta*, 1959, **20** (3), 205–211.—From 0·1 to 6·5 p.p.m. of Os can be accurately determined by measuring the extinction at 555 mμ of the stable violet complex formed at pH 1 to 1·5 (dil. HCl) when aq. 1-naphthylamine-4:6:8-trisulphonic acid soln. (5×10^{-4} *M*) is warmed for $\simeq 45$ min. at 40° with the soln. of osmate. Beer's law is obeyed and the complex has a composition of 2 moles of reagent to 1 mole of Os^{VI}. Since many common ions interfere, an initial separation of Os should be made by distillation from HNO_3 soln. into 0·05 *N* KOH before applying the spectrophotometric procedure. W. J. BAKER

4797. **Tracer investigation of iridium - platinum separation by anion exchange.** D. D. Busch, J. M. Prospero and R. A. Naumann (Princeton University, N.J.). *Anal. Chem.*, 1959, **31** (5), 884–886.—Rapid separation of Ir from Pt (10 mg of each) is achieved on Dowex A-1 anion-exchange resin, as shown by tracers (^{192}Ir, ^{191}Pt and ^{193}Pt). The metals are converted into MCl_6^{2-} and reduction with hydroxyammonium chloride then produces $IrCl_6^{3-}$; 3% of the Pt is also reduced and eluted with the Ir and 0·5% is lost on the column. The purity of the Pt recovered is >99%. Thus ^{188}Ir can readily be separated from its parent, ^{188}Pt. *Procedure*—Add conc. HCl (2 ml) to the soln. containing 10 mg each of Pt and Ir. Evaporate just to dryness, add 9 *N* HCl (2 ml) and 30% H_2O_2 soln. (2 ml), and again evaporate just to dryness. Add 3 *N* HCl (1 ml) and 10% hydroxyammonium chloride soln. (5 drops), heat for 3 min., and transfer the soln. to the Dowex column at 80°. Elute the $IrCl_6^{3-}$ with 9 *N* HCl and the $PtCl_6^{2-}$ with 50% $HClO_4$. J. P. STERN

4798. **Sampling and analysis of bubbles i by mass spectrometry.** J. C. Neerman and Bryan (Sci. Lab., Ford Motor Co., De Mich.). *Anal. Chem.*, 1959, **31** (4, Part I), 5 —The gas content in bubbles ranges from 0· μl-atm. at 25°; relative concn. in the upper can be determined to within 1 or 2%, and a values (down to 0·05 μl) to within 10% true value. Samples $\frac{7}{16}$ in. square and thick are used for the test. The bubble consists of a modified stainless-steel bellows with a pointed steel end-piece attached valve plunger, and is connected throug valves to the spectrometer analyser tube in the spectrometer vacuum system, respe Detailed instructions for operation are including those for the blank determi Carbon dioxide, N, SO_2, A and O were ide the presence of CO and H_2O was not con Pure compounds are used for calibration. P. D. PARR-RIC

4799. **Rational analysis of fireclays.** Worrall (Brit. Ceramic Res. Ass., Queen Penkhull, Stoke-on-Trent). *Trans. Brit. Soc.*, 1959, **58**, 145–153.—A Yorkshire firecl separated into 7 fractions of particle siz 0·075 to > 36 μ and analysed. The conve methods of calculating the rational analysis assumptions that the fireclay mineral is k and that the alkalis are present as felspar are shown to be unsound. A new method i on the "combined H_2O" content (correct organic matter and carbonate) for the co tion of the clay mineral. It is shown that t mineral in fireclays has a lower Al_2O_3 conte kaolinite, the deficiency being made up substitution of Fe_2O_3 and MgO. An formula for the clay mineral is given, whic more consistent results. J. A. Su

4800. **Chemical methods of phase anal metal - ceramic alloys [alloys used for c based on titanium carbide and chromiu titanium diborides.** N. M. Popova, A. F. Pl and L. V. Zaslavskaya. *Zavod. Lab.*, 1959, 271–273.—Various methods applied to the po material (270 mesh) are described. With t carbide, boiling with HCl leaves the carb attacked, but Ni, Co, Cr and Fe, with 0 Ti, dissolve. The carbide phase contains not Ni and Co. Metallic Nb can be extra boiling with HF but its carbide, together wi of Ti, is unattacked. The phase analysis of t carbide for Mo is carried out by electroc dissolution in 5% NaOH soln. Methods of s the borides of Ti and Cr are also described. G. S. S

See also Abstracts—**4659**, Mercaptosucci in inorganic analysis. **4665**, Use of sep columns in analysis. **4874**, Determination cury in biological material. **4875**, Deter of Tl. **4877**, Determination of Cr in blood. Determination of nitrogen oxides in air. Determination of Mo in water. **4997**, Deter of Mo in plants. **5029**, Use of $Ag^{131}I$ in ch graphy of phosphoric acids. **5039**, Dete Mn in aluminium. **5051**, **5052**, Determinati in gases. **5056**, Thermogravimetry of sulph chlorides. **5059**, Determination of impur bismuth. **5061**, Determinations of traces Pb, Bi and Zn.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives, etc.

01. **Analysis for industry [absorption of nitrogen s in carbon and hydrogen analyses].** A. M. G. onald. *Ind. Chemist*, 1959, **35**, 193–195.—view is presented, in which inorganic absor- (*e.g.*, MnO_2, PbO_2, Cu and soln. of oxidants nc. H_2SO_4) and other absorbents (*e.g.*, amino- enzene, diphenylamine, sulphamic acid, am- um sulphamate and trihydroxyammonium phate) are critically discussed and compared. suggested that suitably prepared MnO_2 yields rate results, has a high absorptive capacity s free from troubles in routine analysis.

S. C. I. Abstr.

02. **Determination of nitrogen in some hetero- c compounds.** I. Čepčiansky and L. Chromcová ool of Chem. Technol., Prague). *Chem. Průmysl*, **9** (4), 188–190.—A new modification of dahl's method based on the use of a mixture $_2SO_4$, H_3PO_4, $CuSO_4$ and Se for the mineraliı of the sample permits the determination of isatin and its derivatives (4-chloro-7-methoxy- ı and 4-bromo-7-methylisatin), and in benzo- ole, 1-phenylbenzotriazole and 2-phenylbenzo- ole and their substitution derivatives. Divide xture of 3 vol. of H_2SO_4 (96%) and 1 vol. of O_4 (65%) into two equal parts; to the first add $O_4.5H_2O$ (1 g per 100 ml of mixed acids), to second add powdered Se (1 g per 100 ml of d acids); dissolve both mixtures separately heating, cool, combine and heat to boiling. 0·1 to 0·15 g of sample, 15 to 22 ml of the tion mixture is recommended. J. Zýka

03. **Simultaneous micro-determination of gen and of chlorine and bromine in organic pounds.** A. Konovalov (Univ., Louvain). *Chim. Belge*, 1959, **24** (3), 259–262.—*Procedure* he weighed sample (1 to 5 mg) is placed in a lling flask with 100 ml of water and 2 ml of KOH soln. and a glass bead. The delivery is arranged to be 1 mm from the surface of iquid in the receiver (a 250-ml flask containing l of water and 2 to 5 ml of 0·02 *N* HCl). The d in the flask is distilled quietly (1 hr.) until vol. is reduced to 2 ml. The excess of acid in receiver is titrated with 0·02 *N* NaOH, with rops of Tashiro indicator (2 ml of 1% aq. ıylene blue and 100 ml of 0·04% methyl red 0% ethanol). The residue from the distillation ashed out into a Berlin flask with 10 ml of r, and 2 ml of 3% H_2O_2 is added to convert rate or bromate into halide, and the soln. is d down to 10 ml to destroy excess of H_2O_2. liquid is neutralised to phenolphthalein with HNO_3 (1:2) and 2 ml in excess is added. An ss of 0·01 *N* $AgNO_3$ is added and the soln. tered through a medium comprising a layer of stos (washed with HNO_3) covered with 1·5 cm a sand and 0·5 cm of cotton wool. To the clear ıte are added 1 ml of 0·5% starch soln. and 8 s of 0·02 *N* ethanolic iodine, and the excess of O_3 in this colourless soln. is titrated with 0·01 *N* o a blue colour. Results are given for 18 com- nds, including acid amides, cyanides, amine hydrochlorides and bromine derivatives of acid amides and cyanides. E. J. H. Birch

4804. **Rapid micro-analytical method for the determination of phosphorus and for the simultaneous determination of phosphorus and halogens in organic substances.** W. Merz (Badischen Anilin- & Soda-Fabrik A.-G., Ludwigshafen/Rhein, Germany). *Mikrochim. Acta*, 1959, (3), 456–465.—The sample (3 to 8 mg according to the expected P and halogen content) is wrapped in ashless filter-paper and burned in an oxygen-filled Erlenmeyer flask as recommended by Schöniger (*cf. Anal. Abstr.*, 1955, **2**, 1816). The combustion products are absorbed in a suitable soln. and the Cl^- or Br^- content is determined by the Vieböck method (*Ber.*, 1932, **65**, 496), and I^- by the Leipert method (*Biochem. Z.*, 1933, **261**, 436). The determination of P is carried out photometrically, with the molybdenum blue reaction suitably modified for routine use. An accuracy of $\simeq \pm$ 0·2% is attainable for the P determination and a similar accuracy for the simultaneous determination of P and halogens. The method may be used for semi-micro determinations without modification.

D. F. Phillips

4805. **Gravimetric semi-micro determination of phosphorus as quinoline molybdophosphate in organic compounds.** T. R. F. W. Fennell and J. R. Webb (Royal Aircraft Estab., Farnborough, Hants, England). *Talanta*, 1959, **2** (2), 105–108.—The quinoline molybdophosphate finish is applied to the semi-micro determination of P following fusion with Na_2O_2 (Fennell *et al.*, *Analyst*, 1957, **82**, 639). Drying at 110° causes no decomposition of the ppt., and the theoretical factor (0·01400) is applicable for calculating the P content. Errors due to the presence of 1·5 g of NaCl and 0·02 g of F^- as NaF are insignificant on the semi-micro scale. A two-crucible weighing technique is described which minimises errors due to the hygroscopicity of the ppt. A second method, in which only the working crucible is dried and weighed against a tare, gives only insignificant errors. A third rapid method, in which the crucible after washing is wiped externally, and the ppt. washed with dioxan (3 × 2 ml) and dry diethyl ether (3 × 2 ml) and dried by air suction, is also described. The solvents caused no loss of ppt. It is suggested that the finish could also be used after wet-oxidation or oxygen-flask procedures.

R. E. Essery

4806. **Thermochromic detection of the acetonyl grouping in organic compounds.** E. Sawicki (R. A. Taft San. Engng Center, U.S. Dept. of Health, Education and Welfare, Cincinnati, Ohio, U.S.A.). *Chemist Analyst*, 1959, **48** (1), 4–5.—To 0·1 ml of a methanolic test soln. add 0·5 ml of a 0·5% methanolic soln. of 2-hydroxy-1-naphthaldehyde. Gently agitate the mixture with a stream of HCl gas for 30 sec. Add 0·5 ml of *o*-dichlorobenzene, and boil the mixture vigorously till all the methanol, but none of the dichlorobenzene, has distilled off. Cool the remaining liquid to room temp. A reversible change from a yellow, pink or pale-green colour at the boiling-point to a brilliant green at room temp. indicates a positive reaction. Aliphatic ketones containing the $CH_3{\cdot}COCH_2$– group give a sensitive and specific reaction, the only exceptions being *cyclo*butanone, *cyclo*pentanone and *cyclo*hexanone, which also give a green colour. Other types of carbonyl compounds do not react. The sensitivities for 15 compounds

ranged from 0·2 to 5 μg (phenylpropanone 100 μg). The chemistry of the reaction is discussed.
R. E. Essery

4807. **Determination of straight chains [-$(CH_2)_n$-] of different lengths by infra-red absorption spectroscopy.** A. V. Iogansen (All-Union Sci. Res. Inst. of the Petroleum Ind.). *Zavod. Lab.*, 1959, **25** (3), 302–303.—Saturated hydrocarbons show characteristic bands in the range 720 to 780 cm^{-1}. The non-branching chains can be sub-divided into five structural groups according to the value of n in the chain $-(CH_2)_n-$, thus, (*i*) $n = \not< 6$, (*ii*) $n = 4$ or 5, (*iii*) $n = 3$, (*iv*) $n = 2$, and (*v*) $n = 1$, each of which is characterised by absorption bands of definite frequency, intensity and width. The spectrum of any saturated hydrocarbon can be considered as a superposition of these bands with weights proportional to their relative concn. The analysis according to these structural groups can be carried out in the same way as for a five-component mixture. Group (*v*) is determined from absorption at 782 cm^{-1}, the other four groups show mutual superposition and can be determined by taking measurements at the four points 721, 724·5, 728 and 739 cm^{-1} and solving four simultaneous equations. Examples of the use of the method and confirmation of its validity are given.
G. S. Smith

4808. **Determination of isoprene in reaction mixtures.** M. Kubínová (Res. Inst. Synth. Rubber, Gottwaldov, Czechoslovakia). *Chem. Průmysl*, 1959, **9** (3), 160–164.—A new chromatographic method based on the absorption of isoprene in maleic anhydride has been developed and compared with a colorimetric method based on the formation of a yellow complex after treating the sample with mercuric acetate in anhyd. methanol. It is stated that the chromatographic method gives more precise results over the whole concn. range of the analysed sample. The successful separation of C_5 hydrocarbons has been achieved in a 3-metre column filled with kieselguhr impregnated with ethyl oxo*cyclo*pentanecarboxylate, with H as carrier gas. The method is suitable for mixtures boiling at $\not< 40°$.
J. Zýka

4809. **Analysis of contact gas [formed in the catalytic dehydrogenation of butane, etc.] by a chromatographic method.** V. A. Kolobikhin. *Zavod. Lab.*, 1959, **25** (2), 154–157.—Mixtures of CO_2, H, CH_4, C_2H_6, C_2H_4, C_3H_6, C_3H_8, C_4H_{10} and C_4H_8 are separated and determined by use of heated columns.
G. S. Smith

4810. **Semi-micro method for the characterisation and determination of carbon tetrachloride.** P. Blanc, —. Godfrain and R. Lescure. *Chim. Anal.*, 1959, **41** (2), 54–55.—The method depends on the production of a rose-red colour on treatment of CCl_4 with thymol and copper in alkaline soln. Reagent *A* [100 ml of NaOH soln. (36° Bé) and 10 ml of 5% aq. $CuSO_4$ soln.] (2 ml) is placed in a test-tube (diam. 20 mm) and reagent *B* (5 g of cryst. thymol in 100 ml of 95° ethanol) (2 ml) is added without mixing, followed by 1 ml of a soln. of the sample in 95° ethanol. The tube is fitted with a reflux condenser and immersed in a boiling-water bath for 5 min. After rapid cooling the two phases separate, the alcoholic layer developing a rose-red coloration, the intensity of which depends on the concn. of CCl_4. The reaction is sensitive (1 in $2·5 \times 10^4$) and specific, but the presence of acids, even in the atmosphere, ca interference.
S. M. Mars

4811. **Colorimetric determination of 2-et hexane-1:3-diol.** M. C. Bowman, M. Beroza F. Acree, jun. (Entomology Res. Div., Agric. Service, U.S. Dept. of Agric., Orlando, F *J. Agric. Food Chem.*, 1959, **7** (4), 259–26 *Procedure*—Dilute aliquots (containing 0 to 105 of a standard aq. soln. of 2-ethylhexane-1:3- (**I**) (52·5 μg per ml) in stoppered tubes to with water, cool in ice, and keep cool during addition of 4 ml of reagent [freshly prepared 0 (w/v) soln. of *p*-dimethylaminobenzaldehyde conc. H_2SO_4]. Heat the tube in boiling w (stoppering tightly when warm) for 60 min., in ice for 5 min. and, after 30 min. at 25°, mea the extinction of each soln. at 500 mμ aga water. Plot the extinctions (corrected for reagent blank) against concn. For the determ tion of the insect repellent on glass, cloth or s wash the area with sufficient re-distilled eth (95%) to effect complete extraction and dilu suitable aliquot with enough water to bring concn. of **I** within the range covered by the stanc curve, and the concn. of ethanol to $\not> 5\%$. Dev the colour in a suitable aliquot of the diluted s as in the preparation of the standard curve.
S. C. Joll

4812. **Analysis of dilute aqueous glycerol s tions with quinquevalent vanadium.** D. M. V and D. A. Skoog (Stanford Univ., Calif., U.S. *Anal. Chem.*, 1959, **31** (4, Part I), 586–588.— method is based on the oxidation of glycero formic acid by an acidic soln. of V^V, the exces reagent being back-titrated with a Fe^{2+} soln., N-phenylanthranilic acid as indicator. Results accurate to within $\pm 3\%$ in the concn. range 34 mg of glycerol and compare favourably v those of other methods based on oxidation.
G. P. Coo

4813. **The Molisch reaction.** T. A. Tur (Chem. Dept., Univ. of Auckland, Auckland, Zealand). *Analyst*, 1959, **84**, 194.—The utilit the Molisch reaction can be extended if it is app by adding 0·1 g of the sample to 0·1 g of 1-naph in a dry test-tube, then adding 2 ml of conc. H_2 and mixing. A violet or red - violet colour forn slowly indicates the presence of a compound ha a cyclic ketol group, *e.g.*, a pentose, hexose, p saccharide or glycoside.
A. O. Jone

4814. **Improved method for determination reducing substances [carbohydrates].** V. M. I and M. I. Shoĭkhet. *Sakhar. Prom.*, 1959, (2), 3 Fehling's soln. No. 2 is replaced by a soln. cont ing 128 g of trihydroxyglutaric acid and 207 g NaOH in 1 litre. The procedure remains the s as for the Bertrand method. The quantity reducing sugars is kt, where $k = 0·6068$, 0·6 0·6047 and 0·9383 for glucose, fructose, in sugar and maltose, respectively, and t is the amo of Cu in the copper oxide ppt.
Sugar Ind. Abst

4815. **Use of 4:5-dinitroveratrole for the ul micro determination of reducing sugars on p chromatograms by a reflectance method.** Bevenue and K. T. Williams (W. Utilisation and Devel. Div., Agric. Res. Service, U.S. D of Agric., Albany, Calif.). *J. Chromatogra* 1959, **2** (2), 199–201.—The chromatogram is dip in a 1% soln. of 4:5-dinitroveratrole in acet dried and then dipped in *N* KOH in 95% etha

olour is developed by heating at 60° for 10 n a humid atmosphere and is then measured e paper with a reflectance photometer at u. The plot of extinction *vs.* concn. of sugar ar over the range 2 to 20 μg for fructose, e and arabinose. W. T. CARTER

6. **The hydrolysis of oligosaccharides with to their reductimetric determination.** K. and H. Ruttloff (Inst. Ernährung, Potsdam-ücke, Berlin, Germany). *Ernährungsforschung*, **4** (1), 44–52.—The extent and rate of hydro-f sucrose, lactose, maltose and raffinose by chloric and citric acids are studied by means per chromatography. The formation of lycosidation products, under acid conditions, vn on the chromatograms when the solutions romatographed without prior neutralisation. J. V. RUSSO

7. **Paper chromatography of 2:4-dinitro-hydrazones. Resolution of alkan-2-one, *n*-l, alk-2-enal and alka-2:4-dienal derivatives.** Gaddis and R. Ellis (Dairy and Meat Lab., Dept. of Agric., Beltsville, Md.). *Anal. Chem.*, **31** (5), 870–875.—These derivatives with lengths up to C_{14} can be separated into their ogous classes by ascending chromatography treated paper, with light petroleum (boiling-37° to 40°) as solvent. The individual com-ts of each class can then be separated by chromatographic methods. R_F values are All the derivatives can be identified except of acetone, acetaldehyde, propionaldehyde, ldehyde and acraldehyde. G. BURGER

8. **Separation and identification of *n*-alkanals, 2-ones, alk-2-enals and alka-2:4-dienals by chromatography and direct spectrophotometric nation.** M. Nonaka, E. L. Pippen and G. F. (Agric. Res. Service, U.S. Dept. of Agric., y, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** 75–877.—These compounds can be separated escending chromatography of their 2:4-phenylhydrazones dissolved in $CHCl_3$ on impregnated with a soln. of 2-phenoxy-l in acetone. The developing solvent is *n*-ne. *R* values are referred to the *n*-heptalde-derivative. The spots are cut out and the tion spectra examined in the range 300 mμ mμ. In the chromatogram, compounds of ame homologous series are separated but p with those of a different series and different length. As each homologous series has a teristic absorption max., the individual unds can be identified. Care is necessary nknown mixtures, as compounds outside the studied may cause interference. G. BURGER

9. **Simultaneous determination of formalde-and paraformaldehyde.** Z. Bellen (Anal. Inst. of Gen. Chem., Warsaw). *Chem. Warsaw*, 1959, **4** (1–2), 13–18.—The main lty is due to the decomposition or depoly-tion of paraformaldehyde in the presence D, which reaches a maximum at pH 4 to 5 . The recommended method is based on the nsation of dimedone with formaldehyde, esulting compound being pptd. in acetate at pH 4·6, filtered off and determined by on with standard NaOH soln., without position of paraformaldehyde. The sum of rmaldehyde and polymers is determined by oxidation with iodine in alkaline soln., acidifying and titrating the excess of iodine with $Na_2S_2O_3$ soln. in the presence of starch. The content of paraformaldehyde is obtained by difference. W. B. MIASKOWSKI

4820. **Analysis of formaldehyde. Proposed revision of TAPPI standard T600m-45.** *TAPPI*, 1958, **41**, 170A–171A.—The method is essentially the same as those of the U.S.P. and of the A.O.A.C., the major change being the introduction of bromothymol blue as an alternative indicator, which gives a sharper end-point than litmus. To 50 ml of *N* NaOH add 50 ml of H_2O_2 (3%) and approx. 3 ml of formaldehyde soln. weighed accurately with a weight pipette. Place a funnel in the neck of the flask, heat on a steam bath for 5 min. with shaking, cool and titrate the excess of NaOH with *N* HCl or H_2SO_4, with bromothymol blue or litmus as indicator. The acidity of the formaldehyde is corrected for by a separate titration of 3 ml with NaOH soln. without H_2O_2. The titration of formaldehyde in Na_2SO_3 soln. with H_2SO_4, with aurin as indicator, is recommended for control purposes. R. AICHER

4821. **Determination of aldehydes by the argentimetric method. I. Determination of formaldehyde in technical hexamine.** Z. Bellen and B. Sękowska (Anal. Depart., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 25–28.—The method is based on the oxidation of formaldehyde by means of Tollens reagent prepared from a known quantity of $AgNO_3$ and the potentiometric titration of Ag^+ with a standard soln. of KI. Under these conditions hexamine does not undergo decomposition into formaldehyde and ammonia. The method is sufficiently accurate for technical purposes. W. B. MIASKOWSKI

4822. **Bromimetric determination of acraldehyde in aqueous medium.** J. Młodecka (Anal. Dept., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 53–58.—The method is designed for visual or electrometric determinations in aq. soln., and is based on the titration of a known volume of acidified bromate - bromide soln., standardised with pure acraldehyde, with the test soln. in a closed flask. The end-point is determined either potentiometrically by the dead-stop method or visually by the red colour of methyl orange, added at the moment of the discharge of the bromine colour. The method is short and simple, but speed is essential, since the soln. of acraldehyde is unstable. The error is $\simeq 1{\cdot}5\%$. W. B. MIASKOWSKI

4823. **Analytical chemistry of the aldehydes. II. Iodimetric procedure for the direct determination of glyoxal by means of its bisulphite compound.** E. Schulek and L. Maros (L. Eötvös Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1959, **19** (4), 473–478 (in German).—The glyoxal is converted into its bisulphite compound by adding it in aq. soln. (10 ml, $\simeq 0{\cdot}1\%$) to an excess of Na_2SO_3 soln. (5%) (3 ml) made slightly acid to methyl red with acetic acid. The mixture is covered with a layer of pentane and set aside for 30 min. The excess of SO_2 is then removed with iodine, and 2 ml of hydroxyammonium chloride soln. and 5 ml of 20% NaOH soln. are added. After 5 min. the mixture is neutralised with 20% HCl soln., 2 ml is added in excess, and the liberated H_2SO_3 is titrated with 0·1 *N* iodine. G. BURGER

4824. **Determination of acetone and ethyl methyl ketone in the presence of aldehydes.** L. N. Petrova, E. N. Novikova and A. B. Skvortsova. *Zhur. Prikl. Khim.*, 1959, **32** (6), 1407–1409.—The method of oxime formation can be used if there is a preliminary reaction with aniline to convert aldehydes into non-volatile Schiff bases. *Procedure*—Heat the distillation flask containing $\simeq 0{\cdot}5$ g of an aldehyde - acetone or aldehyde - ethyl methyl ketone mixture and a 15% soln. (5 ml) of aniline in benzene to 50° to 60°; gradually raise the temp. of the paraffin bath to 150°. Collect the distillate in a 0·5 *N* hydroxyammonium chloride soln. (20 ml) in 80% ethanol and, after washing the condenser with ethanol or methanol (15 ml) without disconnecting the apparatus, titrate the HCl in the receiver with a 0·5 *N* ethanolic KOH soln. to bromophenol blue. Determine the amount of aniline distilled over with the benzene in the absence of the test mixture. The presence of H_2O, alcohols, ethers and acids in the test mixture does not affect the determination. K. R. C.

4825. **Colorimetric determination of *n*-amyl ethyl ketone with 3:5-dinitrobenzoic acid.** D. H. E. Tattje and C. Scholtens (Lab. Farm. Galen. Farm., Rijksuniv., Groningen, Holland). *Pharm. Weekbl.*, 1959, **94** (5), 137–142.—An ethanolic soln. (4 ml containing $\gtrsim$ 12 mg of the ketone) is mixed at 20° with 5 ml of an ethanolic 4% (w/v) soln. of the acid, followed by 2 ml of 3·5 *N* NaOH. The colour intensity is measured after 20 min. in a 0·5-cm cell at 545 mμ against a freshly prepared blank. The calibration graph is linear. Values obtained for saponified lavandin and lavender oils are in good agreement with those obtained by the hydroxyammonium chloride method, and show better reproducibility. Values determined directly on the oils are (unaccountably) 50 to 300% higher than those given by the hydroxyammonium chloride method. Cineole, linalol and D-camphor do not react with the acid. P. S. Arup

4826. **Paper-chromatographic identification of organic acids resulting from fermentations.** A. D. Panek (Lab. of Microbiol., Inst. of Chem., Univ. of Brazil, Rio de Janeiro). *J. Chromatography*, 1959, **2** (2), 211–213.—The mixtures of acids were separated by ascending chromatography on Whatman No. 1 paper, with *n*-propanol - conc. aq. NH_3 (7:3) as solvent (*cf.* Isherwood and Hanes, *Anal. Abstr.*, 1954, **1**, 314). After development for 7 hr. the spots were detected by being sprayed with ethanolic bromocresol green soln. and heated at 80° for 10 min. Results are given for the examination of mashes from alcoholic, lactic, acetic and citric fermentations. W. T. Carter

4827. **Automatic derivative potentiometric and spectrophotometric titrations of organic acids.** H. V. Malmstadt and D. A. Vassallo (Univ. of Illinois, Urbana, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 862–865.—Carboxylic acids, sulphonamides, imides, thiols, phenols and enols dissolved in any solvent mixture containing $\lesssim$ 90% of acetone can be titrated with tri-*n*-butylmethylammonium hydroxide. The end-point may be detected by derivative potentiometry or spectrophotometry. Of the electrode combinations tried, none showed any advantage over platinum and graphite (from a 9H pencil). A table of indicators is given for the spectrophotometric method. These indicators can also be used for visual end-point detection. The potentiometric method is not suitable for very weakly acidic materials (*e.g.*, phenol). The pr of each method is within the burette-reading G. Bur

4828. **Oxidation of some organic comp with standard solutions of quinquevalent vana** D. M. West and D. A. Skoog (Stanford Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (4, P 583–586.—α-Hydroxy acids appear to react s eiometrically with an acidic soln. of V**V**, the pr being CO_2 and the corresponding carboxyli with one C atom less than the original subs The excess of vanadate is back-titrated $(NH_4)_2SO_4.FeSO_4.6H_2O$ soln., with N-p anthranilic acid as indicator. The relative e $< 1\%$ for lactic acid in the concn. range o 70 mg. G. P. C

4829. **Analysis of high-molecular-weight q nary ammonium chlorides.** M. Babcock, Terry and A. J. Milun (Chem. Lab., Gen. Inc., Minneapolis, Minn., U.S.A.). *J. Ame Chem. Soc.*, 1959, **36** (3), 93–94.—The meth applicable to the determination of quate ammonium chloride, amine hydrochloride (o alkali) and NaCl in commercial products. *hydrochloride*—The sample (1 to 2 g) is dis in 50 ml of ethanol - toluene (1:1) and ti with either 0·05 *N* NaOH, or 0·5 *N* HCl fo alkali, with phenolphthalein as indicator. *An* The sample (2 g) is dissolved in 50 ml of e and titrated with 0·2 *N* HCl, with bromc green as indicator. *NaCl (in the absence o alkali)*—The sample (1 to 2 g) is ignited, an residue is extracted with 5 to 10 ml of The extract is warmed, then diluted with 15 ml of water, and the soln. titrated with $AgNO_3$, with dichlorofluorescein as indi *Quaternary ammonium chloride*—The sample is dissolved by warming with 50 ml of *iso* alcohol, dichlorofluorescein soln. is added an soln. is titrated with 0·1 *N* $AgNO_3$. G. R. Whal

4830. **Determination of nitrite in quate ammonium nitrites.** J. V. Killheffer, jun., a Jungermann (Armour Chem. Div., Chicago U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 582.—Nitrite ions are oxidised to NO_3^- with amine T, the excess of which is determined metrically. Dimethylformamide is used as so The precision is approx. $\pm 0{\cdot}3\%$ and recoveries pure compounds are 99·8 to 100·2%. G. P. C

4831. **Mass-spectrometric analysis of ali amides.** J. A. Gilpin (Dow Chemical Co., Mi Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 939.—The mass spectra of 35 aliphatic a have been tabulated and correlated. Fragn tion and re-arrangement ions characteris primary, secondary and tertiary amides are as well as spectral characteristics common three groups. Rules are given for the inter tion of the spectra and the identification o amides. G. Bur

4832. **Infra-red and ultra-violet spectra of nitrolic acid and its sodium salt.** D. J. M (British Nylon Spinners Ltd., Pontypool, Wales). *J. Appl. Chem.*, 1959, **9** (4), 201–2 The infra-red spectra of ethylnitrolic and *n*-p nitrolic acid are recorded together with th spectra of the former and its sodium salt. concluded that the ethylnitrolic acid mo

ns a nitro and an azomethine group and is ly hydrogen-bonded. Nitrous oxide, $NaNO_2$ Na acetate are identified among the dissocia- roducts of the apparently mesomeric sodium F. J. M.

3. **New method for the identification of le thiols by paper chromatography.** A. -Guillarmod and P. Ceschini (Batelle Mem. Geneva, Switzerland). *Helv. Chim. Acta*, **42** (3), 713–715 (in French).—Alkylthiols to C_5H_{11}) are identified by descending paper atography of the 2:4-dinitrophenylhydrazones eir 3-alkylthiopropionaldehydes, which are d by the reaction of the thiols with acralde- The stationary phase is dimethylformamide he mobile phase *cyclo*hexane saturated with hylformamide. R_F values are given. G. P. Cook

4. **Titrimetric determination of alkyl mercaptan ie thiol] - dialkyl sulphide and alkyl mercaptan - disulphide mixtures.** B. Jaselskis (Univ. of gan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1959, 5), 928–931.—*Procedure for mixtures of an e thiol and a dialkyl sulphide*—Two equal ots are diluted with ethanol. One is acidified itrated for thiol with iodine soln. The other de alkaline and vinyl cyanide is added. This is acidified and titrated with $KBrO_3$ - KBr The dialkyl sulphide content is calculated the two titrations. *Procedure for mixtures of lkane thiol and an alkyl disulphide*—One ot is titrated with iodine soln. The other is ed by heating under reflux with zinc in a ure of ethanol, acetic acid and HCl, and ed with iodine soln. This method is not cable to tertiary alkyl disulphides. The error 1%. G. Burger

5. **Resolution of isomeric xylenes by gas - l chromatography.** Z. Zlatkis, Su-Yu Ling H. R. Kaufman (Univ. of Houston, Tex., A.). *Anal. Chem.*, 1959, **31** (5), 945–947.— xylene isomers can be separated on a stationary e of 1-chloronaphthalene, dimethylsulpholane mixture of ethylene carbonate and 1-nitro- thalene if one of these liquids is supported on rick which has been treated with aqua regia. brick is heated with aqua regia for 1 hr., ed with H_2O, dil. aq. NaOH soln. and H_2O , dried and screened. Chromosorb can be when treated in the same way. The use of such ally treated fire-brick results in improved resolu- enables finer mesh sizes and longer columns e employed, and reduces pressure drop across column. G. Burger

6. **Infra-red absorptiometric determination of ers of xylene and ethylbenzene in their mixtures.** raczkiewicz-Biernacka and B. Kontnik (Dept. ech. Physics, Inst. of Gen. Chem., Warsaw). . *Anal., Warsaw*, 1959, **4** (1–2), 97–104.—A method for the determination of a quaternary ure of *o*-, *m*- and *p*-xylenes and ethylbenzene i.r. absorption has been developed. The rtical bands used were—8·950 μ, 10·175 μ, 25 μ and 10·375 μ for *p*-, *o*- and *m*-xylenes for ethylbenzene, respectively, and the respec- errors of determination were $\simeq$ 0·6%, 0·3%, and 6·3%. A method of successive approxi- on was used for calculation. *p*-Xylene in the nce of other isomers of xylene can be simply rapidly determined by the application of the base line method, with *cyclo*hexane as solvent, with a possible relative error of 2%. W. B. Miaskowski

4837. **Application of quantitative molecular spectrographic analysis to certain stages in the production of phenol and acetone [from *iso*propylbenzene hydroperoxide].** N. K. Rudnevskii and V. V. Zharkov (Gorki State Univ.). *Zavod. Lab.*, 1959, **25** (3), 297–298.—The determination by i.r. absorption of *iso*propylbenzene hydroperoxide, 2-phenylpropan-2-ol and acetophenone in technical hydroperoxide is described. G. S. Smith

4838. **High-frequency conductimetric titration of weak acids.** G. R. Jamieson (Chem. Dept., Tech. Coll., Paisley, Scotland). *J. Appl. Chem.*, 1959, **9** (4), 209–212.—The conductimetric titration of phenols, enols and imides, in aq. soln., with LiOH soln. and a high-frequency titrimeter is described. Typical titration plots obtained by both direct titration with alkali and back-titration of an excess of hydroxide with standard HCl soln. show sharp breaks at the end-points, and satisfactory results for monohydric phenols, etc., by both procedures are tabulated. Dihydric and trihydric phenols give a curve with two breaks, only one of which is sharp enough for quantitative determinations. F. J. M.

4839. **Chromatographic separation of phenol and cresol isomers. I.** J. Młodecka (Dept. of Physico-Chem. Methods of Analysis, Inst. of Phys. Chem., Polish Acad. of Sci., Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 157–160.—Good results in the separation of phenol and cresol isomers were obtained on Whatman papers No. 1 and 4, and on No. 4 impregnated with phosphate buffer (pH 8), with *cyclo*hexane - $CHCl_3$ - ethanol (9:1:0·6) and benzene - *cyclo*hexane - methanol (2:24:0·25) as solvents and sulphanilic acid to reveal the spots. The components separated in the following order —*o*-cresol, *p*-cresol, *m*-cresol and phenol, giving yellow - brown, purple, orange and yellow spots, respectively; only *p*- and *m*-cresols overlapped. The time required for the analysis on Whatman No. 1 paper is 2 hr., and on the buffered Whatman No. 4 is 45 min. W. B. Miaskowski

4840. **Colorimetric determination of *p*-cresol.** J. Młodecka (Anal. Dept., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 45–52.—The method is based on the formation of a reddish compound by the reaction of *p*-cresol with 1-nitroso-2-naphthol and HNO_3 in glacial acetic acid with subsequent dilution with water. The absorption is measured after 20 min. by means of a Visomat absorptiometer with green filters, a yellow soln. of the reagent being used as reference. The absorption reaches a maximum at 500 mμ, and the method is suitable for concn. of 20 to 500 μg of *p*-cresol per 50 ml. Neither *m*- or *o*-cresol nor phenol interferes. For the visual comparison of the sample soln. with prepared standards the excess of the reagent can be removed after 20 min. by shaking twice with $CHCl_3$. W. B. Miaskowski

4841. **New sensitive test for quinones.** V. Anger (Lab. Dr. Anger, Vienna 18). *Mikrochim. Acta*, 1959, (3), 386–388.—Diphenylbenzidine in 75% H_2SO_4 reacts with *o*-quinones and *p*-benzoquinone to give a blue colour, and forms the basis of a spot-test for microgram amounts of these compounds. D. F. Phillips

4842. **Determination of crotonaldehyde and acetaldehyde in technical crotonaldehyde.** Z. Bellen and N. Bellen (Anal. Dept., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 19–23.—The method described enables both aldehydes to be determined simultaneously. Crotonaldehyde is determined by bromination with a soln. of $KBrO_3$ and KBr in acid medium, the excess of bromine being measured iodimetrically. For the determination of acetaldehyde, both aldehydes are condensed with $NaHSO_3$, and the acetaldehyde liberated by distillation of the product in the presence of $NaHCO_3$ is passed into a known quantity of neutralised hydroxyammonium chloride soln.; the resulting HCl is titrated with a standard soln. of NaOH. The methods are simple, rapid and sufficiently accurate for industrial purposes. Details of an apparatus for the distillation of acetaldehyde are given. W. B. MIASKOWSKI

4843. **Chromatographic analysis of mixtures of benzene polycarboxylic acids.** J.-E. Germain, J. Montreuil and P. Koukos (Centre de Carbochimie, Fac des Sci., Lille, France). *Bull. Soc. Chim. France*, 1959, **26** (1), 115–120.—The benzene polycarboxylic acids obtained by oxidising coal are separated by paper chromatography on Whatman No. 1 paper by the descending technique. The solvent system *iso*propyl alcohol - formic acid - aq. phenol (950 g of phenol in 100 ml of water) (1:2:17) separates the acids into groups of isomers. The system ethanol - aq. NH_3 (*d* 0·925) (7:3) separates benzoic and phthalic acids from the mixture of *iso*- and tere-phthalic acids. The system ethanol - conc. aq. NH_3 (1:1) separates tri- and higher poly-carboxylic acids (except benzene-1:2:3:4-tetra- and 1:2:4:5-tetra-carboxylic acids. The system butanol - acetic acid - water (4:1:5) separates tri- and higher poly-carboxylic acids (except -1:2:3-tri- from -1:2:4-tri- and -1:2:3:5-tetra- from 1:2:4:5-tetra-carboxylic acid). A complete separation of tricarboxylic and higher acids therefore can be made by two-dimensional chromatography. The spots, detected as dark spots against the blue fluorescence of the paper under u.v. light, can be cut out and extracted with 0·01 *N* NaOH and the quantity determined by u.v. absorption. The u.v. spectra of the acids in alkaline soln. are given. The synthesis of the pure acids is outlined. E. J. H. BIRCH

4844. **Chromato-polarographic studies. XIII. Analysis of mixtures of aliphatic nitro compounds.** W. Kemula and D. Sybilska (Dept. of Physico-Chem. Anal. Methods, Inst. of Phys. Chem., Polish Acad. of Sci, Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 123–134.—The chromato-polarographic method for separation of the homologous nitroalkanes is based on reversed-phase partition chromatography, with either rubber or cellulose acetate as a carrier for the liquid stationary phase. The use of cellulose acetate involves not only partition, but also adsorption on the carrier. The method covers the quant. and qual. analysis of a mixture consisting of the first six nitro derivatives of the methane series, and their separation can be carried out in the presence of 1:2-dinitroethane and 1:3-dinitropropane. The same technique can be used for the separation of higher homologues of this series. Details are given of quant. determinations by both elution and frontal methods, together with examples of the results, with emphasis on the elution procedure. The error is $\gtrsim 5\%$. The frontal method has proved accurate only in the separation of the first th four components of the series.

W. B. MIASKOW

4845. **Chromato-polarographic analysis of aniline mixtures. I. Choice of the syste solvents.** H. Buchowski and W. Pawłowski (of Inorg. Chem., Warsaw Univ.). *Chem. Warsaw*, 1959, **4** (1–2), 135–143.—The difficu separating *m*- and *p*-nitroaniline lies in similar ionisation constants and partition coeffi when a water - toluene solvent system is empl The best conditions are obtained when sol strong acids are used as eluents. The substit of a mixture of $CHCl_3$ and acetone for to increases the partition coefficients, and con separation of *m*- and *p*-nitroaniline can be obt on a column 4 cm high.

II. Quantitative determination. H. Buch and W. Pawłowski. *Ibid.*, 1959, **4** (1–2), 145–1 The frontal method is used, with a 4-cm co packed with 100 mg of rubber. The aq. pha a mixture of 1000 g of 3% aq. H_2SO_4, 166·5 acetone and 88·3 g of $CHCl_3$ is used as eluen the $CHCl_3$ phase for swelling the rubber. All isomeric nitroanilines can be determined di from a chromato-polarogram if 20 ml of elu collected. The analysis can be accomplished more rapidly if only the *m*- and *p*-nitroanilin determined from an initial 7 to 8 ml of eluate the *o*-isomer obtained by difference from the amount of nitroanilines determined polarogr cally. In order to obtain reproducible result necessary to plot calibration curves and carr all analyses under strictly standard condi The time required for complete analysis is < and the mean error is $\gtrsim 1\%$.

W. B. MIASKOW

4846. **Fluorimetric micro-determination of phenylboron.** Anil K. Mukherji and Bhara Sant (Coates Chem. Lab., Louisiana State U Baton Rouge, U.S.A.). *Mikrochim. Acta*, (3), 370–371 (in English).—Preliminary e ments indicate that the sparingly soluble yello white compound $(C_6H_5)_4B(C_9H_7ON)_2$ forme the interaction of 8-hydroxyquinoline (**I**) an tetraphenylboron (**II**) (*cf.* Crane, *Anal. C* 1956, **28**, 1794) exhibits a characteristic fluorescence under u.v. light which may be for the micro-identification of $B(C_6H_5)_4^-$. *cedure*—Place single drops of reagent (2% of glacial acetic acid), glacial acetic acid and soln. on a filter-paper and dry carefully. E to u.v. light. Alternatively, instead of a acetic acid separately to the paper, use a so **II** acidified with acetic acid. To mask the io Be, Mg, Ca, Zr^{IV}, Al, Zn and Cd, which also duce fluorescence with **I**, add NaF. The lim detection is 1 μg of **II**. D. F. PHILL

4847. **Titrimetric determination of tetraph boron by thallium.** Bharat R. Sant and An Mukherji (Coates Chem Lab., Louisiana Univ., Baton Rouge, U.S.A.). *Talanta*, 19 (2), 154–155.—Sodium tetraphenylboron (**I**) is treated with a known excess of standard T soln. and set aside for 4 to 6 hr. The ppt. is filtered off on a Whatman No. 42 paper, wash to 10 times with about 10 ml of water, and t in the filtrate titrated with standard KIO_3 in 3 *N* to 4 *N* HCl to the ICl end-point, $CHCl_3$ as indicator. The difference betwee blank and assay titres gives the amount of **I** of *M* KIO_3 soln. ≡ 0·68447 g). The rel

racy is ≃ 1% for amounts of **I** from 0·03 to g. Soln. of **I** are unchanged after storage in rigerator for 7 days, but when stored in day- at 30° a decrease in concn. of **I** occurs after r. R. E. Essery

48. **The longest wavelength band in the ronic spectra of polycyclic aromatic hydrocarbons analytical use.** C. Karr, jun. (Low-Temp. Tar , Bureau of Mines, U.S. Dept. of the Interior, gantown, W. Va.). *Appl. Spectroscopy*, 1959, **13** 15–25; (2), 40–45.—A table is presented listing polycyclic aromatic hydrocarbons, with 3 or more natic rings, according to increasing values in of the longest wavelength absorption band. A edure is recommended for using this table in analysis of samples, such as those obtained in tionation by elution chromatography.
K. A. Proctor

49. **New colour tests for the larger polynuclear natic hydrocarbons.** E. Sawicki and R. Barry A. Taft San. Engng Center, U.S. Dept. of lth, Education and Welfare, Cincinnati, Ohio). *nta*, 1959, **2** (2), 128–134.—Colour tests are ribed for polynuclear aromatic hydrocarbons terest in the study of air pollution by carcino- . The reagents are prepared in a specified ner with PCl_5 and furfuraldehyde, 2-thenalde- e, 4-dimethylamino-3-nitrobenzaldehyde (**I**), 9- raldehyde (**II**) and indol-3-ylacetaldehyde. l ml of test soln. are added 1 ml of reagent, ml of $POCl_3$ and trifluoroacetic acid to a vol. 0 ml, shaking after each addition, the spectrum g then determined against a solvent - reagent k. Wavelength maxima and sensitivities are n for the colours given by these reagents with umber of aromatic hydrocarbons. Of the ents studied, **II** gave intense bands in the on 800 to 960 mμ; **I** also reacted with the more c polynuclear hydrocarbons, but gave negative lts with a number of simple aromatic hydro- ons which react with piperonylidene chloride. acts readily in most cases with the benzene act of air-particulate samples, and is ideal for analysis of *peri*-condensed aromatic hydro- ons. Trifluoroacetic acid can be used in place Cl_5 in preparing the reagent, but no advantage ined thereby. R. E. Essery

50. **Ultra-violet absorption spectra of 8- roxyquinoline and its copper chelate.** Kumar hna Chatterjee (Univ. Coll. of Sci., Calcutta, a). *Anal. Chim. Acta*, 1959, **20** (3), 232–235.— 5% ethanol soln. 8-hydroxyquinoline shows rption maxima at 242 mμ and 310 mμ, but ethanolic 0·1 *N* KOH the band at 242 mμ s to 256 mμ, whilst in ethanolic 0·1 *N* $HClO_4$ free ligand shows absorption maxima at 255, 320 and 370 mμ. The Cu oxinate in ethanol shows two maxima at 258 and 395 mμ, the mμ band of the ligand having shifted to 395 mμ. n these observations a probable structure for Cu oxinate molecule is suggested.
W. J. Baker

51. **Separation and identification of pteridines paper chromatography.** H. Kwietny and F. gmann (Dept. of Pharmacol., Hebrew Univ., assah Med. Sch., Jerusalem, Israel). *J. Chroma- aphy*, 1959, **2** (2), 162–172.—The pteridines e separated by descending chromatography on atman No. 1 paper. Several solvent systems e examined of which the most useful was ropyl alcohol - dimethylformamide - 25% aq. NH_3 (13:5:2), but no single solvent could be used to separate all the pteridines studied. The separated compounds were detected with u.v. light and could be determined by u.v. spectrophotometry after extraction from the paper with phosphate buffer (pH 8). The recoveries were in the range 50 to 90%. The dependence of R_F value on the composition of the solvent and the structure of the pteridines is discussed. W. T. Carter

4852. **Determination of 5-acetamido-1:3:4- thiadiazole-2-sulphonyl chloride in the technical product.** N. Bellen and M. Szelągowska (Anal. Lab., Inst. of Gen. Chem., Pharmaceut. Inst., Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 9–11.—The sum of chloride ion and chlorine combined as sulphochloride in a sample hydrolysed by the addition of HNO_3 (1:1) in the presence of diethyl ether is determined potentiometrically by titration with standard $AgNO_3$ soln. The free Cl^- are then determined separately by extracting the sample with H_2O in the presence of diethyl ether, and titrating the acidified soln. with 0·1 *N* $AgNO_3$ as described above. W. B. Miaskowski

4853. **Determination of arsenates in solutions of sodium methylarsonate and dimethylarsinate.** D. Malysz (Inst. of Drugs, Warsaw). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 73–81.—Paper chromatography is employed to separate the components, and the determination of the stage of decomposition is carried out colorimetrically. Arsenates are eluted after separation, and determined in the form of arsenomolybdenum blue. Methylarsonate and dimethylarsinate interfere to a small extent, but the method is suitable for the determination of the products of decomposition to within 10% and is sufficiently accurate for pharmaceutical purposes. W. B. Miaskowski

4854. **Gas-chromatographic determination of fuel dilution in lubricating oils.** R. S. Porter and J. F. Johnson (California Res. Corp., Richmond, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 866–869.—A gas chromatograph is fitted with an inlet system consisting of passages in a block heated to 315°. When the sample, containing dodecane as an internal standard, is injected the volatile fractions are flashed off and swept by the carrier stream (He) through a short column kept at 175° (which traps entrained oil) into the analysing column. This method is more precise and more sensitive than the ASTM method, and samples as small as 0·25 ml can be used.
G. Burger

4855. **Gas-chromatographic determination of dissolved oxygen in lubricating oil.** P. G. Elsey (Ethyl Corp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 869–870.—An inlet system is described which consists of a removable U-tube fitted with two sintered glass discs and an inlet port midway between them. Dissolved gases are removed from the sample contained in this tube by the carrier gas (He) and pass into a column packed with Linde molecular sieve type 5A which separates the components. The limit of detection for O is 0·01% by vol. The standard deviation is 2·2% of the mean. The analysis time is 10 min. and the only known interference is from A, which has the same retention time as O. G. Burger

4856. **Hydrocarbon compound-type analysis of automobile exhaust gases by mass spectrometry.** D. M. Coulson (Stanford Res. Inst., Menlo Park, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 906–

910.—Mono-olefins cannot be distinguished from cycloparaffins by their mass spectra. Separation can be achieved by passing an aliquot of the sample through a column of $Hg(ClO_4)_2$ and $HClO_4$ supported on fire-brick. With a carrier-gas flow of 110 ml per min. at 100°, olefins are quant. absorbed. The mass spectra of treated and untreated aliquots are compared. The interpretation of the spectra is discussed. G. BURGER

4857. **Investigations on *Monarda fistulosa* L. III. Colorimetric determination of thymoquinone.** E. Balcarówna and B. Borkowski (Zakład Botan. Stosowanej i Aklimatyzacji). *Biul. Inst. Roślin Leczniczych*, 1959, **5** (1), 21–26.—Earlier methods of analysis of the essential oil of this plant, containing carvacrol, thymoquinone (**I**) and thymoquinol (**II**), permitted the determination of phenols only, and consequently a new method for the determination of **I** was developed. It is based on the reaction with ethyl cyanoacetate in the presence of ethanol and conc. aq. NH_3. The sapphire-blue colour thus produced is stable for 1 hr. and shows a maximum absorption at 610 mμ with the use of filter S61. The extinction curve is a straight line for concn. from 0·06 to 0·2%. Although the colour reaction with ethyl cyanoacetate had been considered specific for quinones, it was found that **II** produced an identical colour under the same conditions, but this compound is unstable (being oxidised to **I**) and renders the method inapplicable to the direct determination of **I**, so that **II** must be removed by fractionation. The error is ⩽5%. W. B. MIASKOWSKI

4858. **Standard methods for testing filter-paper. Introduction of the tentative standard DIN 53136: Determination of ash.** C. Bräutigam. *ChemikerZtg*, 1959, **83** (5), 158–160.—The complete text of "DIN 53136: Determination of ash" is quoted. The results of a preliminary critical evaluation of various tests for the measurement of other important properties of filter-paper which it is proposed to standardise are described. It is suggested that a modification of the method described in the U.S.A. Specification UU–P–236 for the determination of filtration rate is the most suitable for standardisation purposes, and that wet strength can be satisfactorily measured with the Herzberg apparatus or a simplified modification thereof. Separation ability can be determined by observing the retention of various ppt. [*e.g.*, $Fe(OH)_3$, $PbSO_4$ (pptd. at 80°), $BaSO_4$ (pptd. cold), and Ca oxalate] according to the quality of the paper under test. Acid- and alkali-resistance should be determined with HCl or KOH at 20° for 10 min., the concn. of the soln. depending on the quality of the paper. S. M. MARSH

4859. **Determination of lignin, cellulose and pectin materials in flax and hemp fibres.** C. Săndulescu and I. Raiu (Inst. Agron. Res.). *Acad. R.P.R., Stud. Cercet. Chim.*, 1959, **6** (3), 489–499.—*Lignin*—The method used is that of Ritter *et al.*, modified by Hölder, and further modified by the authors. The material is extracted in a Soxhlet apparatus with ethanol for 4 hr., washed with ethanol, extracted for 3 hr. with water on a water bath under reflux, washed with boiling water and ethanol and air-dried. About 1 g of extract is treated slowly with 15 ml of H_2SO_4 (72%) at 12° to 15° with continuous agitation. After 2 hr. the contents are diluted with water to 3% acid concn., then boiled for 4 hr. on a sand bath under reflux. The ppt. is filtered off, washed with water dried at 105°. *Cellulose*—The method use based on that of Kürschner and Hoffer modifie Ermakov and Arasmovici. The sample (≃1 boiled under reflux for exactly 1 hr. with a mixtu 5 ml of conc. HNO_3 and 20 ml of 96% eth The liquid is decanted off and the residue b again under the same conditions for 30 min., the liquid again decanted. The residue is wa with ethanol by decantation and boiled for 30 in 25 ml of NaOH (1·25%) in a brine bath (NaCl) and filtered. The ppt. is washed with bo water, ethanol and ether, and dried for 2 h 105°. *Pectin materials*—The method used is b on the Nanji and Norman procedure. The sa (≃1 to 1·5 g so that the final ppt. is between and 0·05 g) is extracted with 200 ml of ammo oxalate soln. (0·5%) for 24 hr. in a thermostat at filtered hot and diluted to 250 ml with ammo oxalate soln. A portion (100 ml) is evaporate 20 to 25 ml, the pectin is pptd. with 90 ml of ethanol (acidified with HCl), so that the alcohol concn. is ≃70%, and filtered. The pp washed with ethanol to eliminate oxalate dissolved in very dil. boiling aq. NH_3. The is treated with 0·1 *N* NaOH to obtain pectic and after several hours 50 ml of *N* acetic plus 50 ml of *M* $CaCl_2$ is added and the mixtu set aside overnight. The Ca pectate is coagul by heating, filtered off while hot, washed with water, evaporated on a water bath and drie 105°. H. SH

4860. **Qualitative chromatographic identific of mildew inhibitors on military fabrics.** T Miles and A. C. Delasanta (U.S. Army Qua master Res. and Engng Center, Natick, M *Amer. Dyest. Rep.*, 1959, **48** (7), 31–32.— fabric (5 g) is boiled with 200 ml of CCl_4 unti vol. is reduced to 100 ml. The soln. is chron graphed on a circular paper, butanol - wa conc. HCl (8:1:1) being used to separate C hydroxyquinolinate, Cu naphthenate, di-(5-ch 2-hydroxyphenyl)methane and the zinc sa dithiocarbamic acid, and 2·5% aq. NaOH s ethanol (17:3) for salicylanilide. For the c development, the paper is exposed to NH_3 fu and examined under u.v. light. It is then c half, one half being wetted with a 0·01% sol dithizone in CCl_4 and then dried, the other being wetted with a 2% soln. of 4-aminophena in 0·025% Na_2CO_3 soln., then with 8% $K_3Fe(CN)_6$ soln., and then exposed to NH_3 fu R_F values and colours are tabulated. S.C.I. ABS

4861. **Determination by means of the infr spectrometer IKS-12 of products obtained in c lactam manufacture.** L. G. Zelenskaya, A Iogansen and G. A. Kurkchi (State Sci. Res. Devel. Inst. of the Nitrate Ind. and Produc Org. Synthesis). *Zavod. Lab.*, 1959, **25** (3), 300.—The determination of the hydrogenation nitration products of benzene and of various are referred to. G. S. SMI

4862. **Mass-spectrometric determination of composition of raw materials and interme in the production of caprolactam.** V. Čermák V. Hanuš (Inst. Phys. Chem., Acad. Sci., Pra *Chem. Průmysl*, 1959, **9** (5), 235–237.—It is cla that by means of a mass spectrometer it is pos to determine the content of homologues impurities of higher mol. wt. in concn. ≃ 0

The accuracy of the analysis depends on preliminary treatments by means of the influence of the adsorptive properties test samples is removed or decreased. With strument used the following determinations e carried out: *o*-cresol in phenol, phenol in exanol or *cyclo*hexanone, *cyclo*hexanol in exanone, methyl*cyclo*hexanone in *cyclo*hexor *cyclo*hexanone, methyl*cyclo*hexanol in exanol or *cyclo*hexanone, methylcaprolactam prolactam or *cyclo*hexylidene*cyclo*hexanone. ighest accuracy was reached in determining l*cyclo*hexanone in *cyclo*hexanone (1 to 2% nd the lowest in determining *o*-cresol in exanol (10 to 20% rel.). The ionic source mass spectrometer is connected by a capillary o a 3-litre reservoir into the neck of which sed two tubes, one of which is connected to cury diffusion pump through a valve packed metallic indium and the other contains a ed-glass disc covered with mercury. The e to be analysed is sucked into the exhausted oir through a capillary tube in contact with ntered glass disc below the surface of the ry. J. ZÝKA

3. **Determination of maleic acid in its copoly- with vinyl chloride and vinyl acetate.** P. se, T. Di Gioacchino and E. Garzuglia (Inst. he "Polymer", Terni, Italy). *Chim. e* 1959, **41** (3), 218–221.—The polymer is ved in pyridine in the presence of small nts of water, and maleic acid is titrated with Na methoxide in methanol - benzene, or NaOH in *iso*propyl alcohol, the end-point detected with thymol blue or phenol- lein, or potentiometrically. C. A. FINCH

4. **Chlorine in bleached shellac.** O. Tweet, . Miller and R. H. Simon (S. C. Johnson & Inc., Racine, Wis., U.S.A.). *Soap, N.Y.*, **35** (3), 95–99.—Methods for the determination al Cl and Cl as chloride are outlined and dis- l. Chloride is often incompletely extracted aking the ground sample with water, as the e of extraction depends on the size of the les. The following procedures are recom- ed. *Chloride*—The sample (1 g) is dissolved utanol and the Cl^- are extracted with water. aq. extract is strongly acidified with HNO_3, he Cl^- are titrated potentiometrically with N $AgNO_3$. *Total chlorine*—(*i*) The sample is dissolved in *iso*propyl alcohol, and reduced ating under reflux with metallic sodium (2·5 g). cooled liquid is diluted with water, acidified HNO_3, and an excess of 0·1 N $AgNO_3$ is added, back-titrated with 0·1 N KSCN. (*ii*) The e (0·3 g) is decomposed in a Parr bomb, the is dissolved in water, acidified with HNO_3, the Cl^- are titrated potentiometrically as bed above. The Parr bomb method is probably ly more accurate, but the reduction method re rapid, and needs no special equipment. S.C.I. ABSTR.

5. **Methods of analysis for tall-oil products.** errlinger (Arizona Chem. Co., Panama City, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1959,), 119–124.—A review is presented of the pal U.S. standard methods (mainly A.S.T.M.) ne chemical and physical analysis of tall-oil cts. G. R. WHALLEY

4866. **EDTA titration of copper in copper linoleate.** C. A. Lucchesi, J. A. Stearns and C. F. Hirn (Anal. Res. Dept., Sherwin-Williams Co., Chicago, Ill., U.S.A.). *Chemist Analyst*, 1959, **48** (1), 9–10.—Treat the sample (0·15 g) with 10 ml of HNO_3 (1:3) and heat to boiling for 10 to 30 min., till all the Cu is in solution. Filter into a 250-ml beaker and wash the filter well with hot water, not allowing it to run dry. Add aq. NH_3 (1:3) to the filtrate till a blue or green colour appears, then continue the addition dropwise to pH 7 (indicator paper). The mixture should then measure about 125 ml. Add 1 ml of glacial acetic acid and 6 drops of 0·1% 1-(2-pyridylazo)-2-naphthol soln. as indicator and titrate with 0·01 M EDTA (disodium salt) to a yellow - green colour. To facilitate recognition of the end-point, use a comparison soln. of 6 drops of indicator soln. in 125 ml of acidified water. Duplicate determinations occupy 1 hr., and results show good replication and close agreement with results by the electro-deposition procedure. R. E. ESSERY

4867. **Polarographic determination of dibutyl phthalate in propellent compositions containing nitroglycerin.** A. F. Williams and D. Kenyon (Res. Dept., I.C.I. Ltd., Nobel Div., Stevenston, Scotland). *Talanta*, 1959, **2** (1), 79–87.—It is not possible to determine dibutyl phthalate in ether extracts of explosives because of interference from nitroglycerin, but the determination can be carried out with a cathode ray polarograph. Nitroglycerin enhances the height of the dibutyl phthalate peak, but this effect becomes constant at approx. 80 μg of nitroglycerin per ml, above which a blank value can be deducted. *Procedure*—Extract the sample (1 g) with diethyl ether for 6 hr. Evaporate the extract to dryness and dilute to 100 ml with methanol. Dilute 10 ml of this soln. to 100 ml with methanol and take a 10-ml aliquot. Add tetramethylammonium iodide soln. (0·2 M) (10 ml), remove O by passing N for 10 min. and polarograph, measuring the peak height at $-1{\cdot}52$ V (mercury pool). Carry out a blank on a soln. of nitroglycerin containing $\lessdot$ 80 μg per ml. Results are in good agreement with the dibutyl phthalate content found by difference when a complete analysis procedure is used. W. T. CARTER

See also Abstracts—**4658**, Schiff's reagent of calibrated sensitivity. **4722**, Determination of N. **4724**, Determination of aniline in hydrazine. **4726**, Phosphorus in coal and coke. **4899**, Determination of quinolinic acid. **4962**, **4963**, **4964**, **4965**, **4966**, Dyes in foods. **4981**, Detection of carboxylic acids and aldehydes in air-borne particles. **5017**, Determination of H_2O in textiles. **5029**, Use of $Ag^{131}I$ in chromatography of organic acids. **5033**, Gas chromatography of organic compounds.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

4868. **Analysis of gas in biological fluids by gas chromatography.** L. H. Ramsey (Dept. of Med., Vanderbilt Univ., Nashville, Tenn., U.S.A.). *Science*, 1959, **129**, 900–901.—The permanent gases in the sample fluid, *e.g.*, plasma (1 ml), are extracted

in a Van Slyke volumetric vacuum extraction chamber, and injected into the carrier stream (He) of a gas chromatograph (Beckman GC-2). The method is illustrated by the determination of oxygen tension in plasma.

H. F. W. KIRKPATRICK

4869. **Radiometric determination of sodium in blood serum.** J. Dobrowolski and N. Wyszyński (Med. Acad. Szczecin). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 207–212.—The method consists in measuring the intensity of the radiation produced by the uranyl - sodium complex by means of a Geiger - Müller counter. The ppt. from this determination was weighed to compare the results obtained by radiometric and gravimetric methods, and it was found that the accuracy of both is of the same order. The error of determination is $\geqslant 4\%$.

W. B. MIASKOWSKI

4870. **Rapid micro-method for determining serum calcium.** I. M. Bett and G. P. Fraser (Dept. of Clin. Chem., Univ. of Aberdeen, Scotland). *Clin. Chim. Acta*, 1959, **4** (3), 346–356.—The proposed method consists in titrating the Ca in serum (0·1 ml) directly with EDTA soln. by using a micro-syringe, with calcein as indicator. The end-point is determined by observing the disappearance of fluorescence in u.v. light. Suitable apparatus is described.

H. F. W. KIRKPATRICK

4871. **Ultra-micro determination of serum calcium.** P. Cartier and J. Clement-Métral (Fac. de Méd., Paris, France). *Clin. Chim. Acta*, 1959, **4** (3), 357–363.—The method is similar to that of Bett and Fraser (*Anal. Abstr.*, 1959, **6**, 4870), the disappearance of the fluorescence during the titration being automatically recorded.

H. F. W. KIRKPATRICK

4872. **Determination of copper and zinc in biological material by activation analysis.** H. J. M. Bowen (Technol. Irradiation Group, Isotope Div., Wantage Radiation Lab., Grove, Berks., England). *Intern. J. Appl. Radiation and Isotopes*, 1959, **4** (3–4), 214–220.—Tomato seeds and blood were irradiated with standards containing $\simeq 0{\cdot}1$ μg of Cu and 20 μg of Zn in a thermal neutron flux of 10^{12} n per sq. cm per sec. for 12 hr. The subsequent separation procedures for the Cu and Zn are described; they take about 2 hr. and 2·5 hr., respectively. It is estimated that 4×10^{-10} g of activated Cu or 5×10^{-8} g of activated Zn would produce a count-rate equal to the background count-rate of an end-window counter.

G. J. HUNTER

4873. **Micro-determination of zinc in blood and other tissues by neutron activation analysis.** T. E. Banks, R. Tupper, E. M. A. White and A. Wormall (Dept. of Biochem. and Physics, Med. Coll., St. Bartholomew's Hosp., London). *Intern. J. Appl. Radiation and Isotopes*, 1959, **4** (3–4), 221–226.—The sample was dried and then irradiated for 28 hr. in BEPO with standards containing 2 μg and 4 μg of Zn and one of 20 to 30 mg of $ZnSO_4$ that was used to facilitate calibration of the counting equipment. The radiochemical separation of the Zn and the subsequent γ-counting of the 14-hr. ^{69m}Zn are described. The mean recovery of Zn (0·78 to 2·0 μg) added to seven samples of blood (0·15 to 0·28 g) was 101%. The content of Zn of mouse liver was found to be $27{\cdot}0 \pm 0{\cdot}6$ μg in 5 determinations; a chemical method gave a content of 28·4 μg.

G. J. HUNTER

4874. **Determination of traces of merc biological material by catalytic action of m ions. Micro-electrolysis of mercury.** D. P and S. Ašperger (Inst. of Inorg., Anal. and Chem., Fac. of Pharm., Univ. of Zagreb, slavia). *Anal. Chem.*, 1959, **31** (5), 939- Ash the sample of blood (5 ml) or urine (25 the presence of HNO_3, sublime the mercu stream of Br vapour and absorb it in b water. Electrolyse the soln. between pl electrodes, re-dissolve the deposited merc bromine water and expel the excess of evaporation. Alternatively, use a gold c and dissolve the deposit in conc. HNO_3. Det the concn. of Hg^{2+} in the purified soln. catalytic effect on the reaction of $K_4Fe(CN$ nitrosobenzene at pH 4·1 to give a violet-c product (*cf.* Ašperger and Murati, *Anal.* 1954, **1**, 1789). Quantities of 0·002 to 0·050 of Hg can be determined with a coeff. of va of $\simeq \pm 20\%$.

A. R. Ro

4875. **Determination of thallium in urine a** E. E. Campbell, M. F. Milligan and J. A. L (Los Alamos Sci. Lab., N. Mex.). *Amer Hyg. Ass. Quart.*, 1959, **20**, 23–25.—Recove Tl average 96 $\pm$ 6%. Procedures are given preliminary preparation of samples, for the p tion of the standard curve, for the oxida thallous ion to thallic ion, and for the ext and measurement of the Tl - methyl violet co

NUCL. SCI. AB

4876. **Polarographic determination of le urine in the presence of EDTA with use of exchanger to remove interfering substance** Chmielowski and Z. Myslak (Clin. Dept., Inst. in Mining and Metall. Ind., Zabrze). *Anal., Warsaw*, 1959, **4** (1–2), 233–239.—A graphic method for determining Pb in the of patients suffering from plumbism and with salts of EDTA consists in acidifying the sample (to pH < 1) with 2 ml of 8% passing the soln. through a column (15 c Wofatit P (H form) to adsorb interferin stances, bubbling CO_2 through the percola 2 min., suppressing frothing with a trace ethylhexanol, and then examining the soln. graphically. Concn. of Pb >30 μg per 100 be determined. With concn. >50 μg per the error is $\simeq \pm 6{\cdot}5\%$.

W. B. MIASKO

4877. **Spectrophotometric determination chromium in human plasma and red cells.** Miller and J. H. Yoe (Dept. of Chem., U Virginia, Charlottesville, U.S.A.). *Clin. Acta*, 1959, **4** (3), 378–383.—The method is on the red - violet complex formed by the re of dichromate with diphenylcarbazide. The of Cr found in normal human plasma range 0·017 to 0·052 p.p.m., and in red cells from to 0·038 p.p.m.

H. F. W. KIRKPAT

4878. **Estimation of iodine in urine.** G. Wi and K. Claeson (Clin. Lab. Centrallasa Danderyd, Sweden). *Acta Chem. Scand.*, 19 (3), 606–607 (in English).—Calcium cause results owing to loss of CaI_2 during incine This can be avoided by a preliminary treatm the urine with ammonium oxalate to remo The sample is then incinerated with Zn a and K_2CO_3 for 3 hr. at 600°. The residue solved in N HCl and the iodine determined metrically by the arsenite - ammonium cer

method. The average recovery of iodine is ompared with 83% when Na_2CO_3 is used for cineration. W. T. CARTER

). Combustion of body fluids in oxygen, and plication to the determination of iodine in containing contrast media. H. Langecker ing A.-G., West Berlin). *Klin. Wochschr.*, **37** (8), 471–472.—Urine or bile (0·5 ml) is ed on a filter-paper cone, and dried at 60° . The paper is then burnt in a flask con- g oxygen, the products of the combustion absorbed by a carbonate - nitrate soln. oxidation of the resulting soln. with bromine, date content is determined by titration with rd $Na_2S_2O_3$ soln. This method gives recoveries o 104% over a range of 6·7 to 670 mg of iodine 0 ml. Owing to the production of peroxides, ethod cannot be used for the determination rum-protein-bound iodine by the ceric - te catalytic reduction method.

D. B. PALMER

). Spectrophotometric determination of NN′- icotinoylhydrazine (DINH) in the presence of id and its metabolites. B. P. Lisboa rkulose-Forschungsinst., Borstel über Bad oe). *Naturwissenschaften*, 1959, **46** (3), 109.— n. (5 ml, containing 25 to 500 μg) of these nces in 0·067 *M* phosphate buffer (pH 7·2) ted with 0·5 ml of a freshly prepared 0·25% oln. of trisodium pentacyanoamminoferrate ferington, *Analyst*, 1953, **78**, 174). After heated in a water bath at 60° for 15 min. the s cooled and the extinction is measured at μ against a blank. DINH shows a selective tion max. at this wavelength and its concn. e obtained from a standard curve.

H. F. W. KIRKPATRICK

1. Improved glucose oxidase method for deter- g blood, cerebrospinal fluid and urine glucose V. Marks (Inst. of Neurol., Nat. Hosp., n, England). *Clin. Chim. Acta*, 1959, **4** 95–400.—The method of Middleton and hs (*cf. Anal. Abstr.*, 1958, **5**, 3087) is modified substitution of an acetate for the phosphate soln. This is prepared by mixing 3 vol. of *M* acetic acid with 7 vol. of 0·15 *M* Na acetate djusting the pH to 5·0.

H. F. W. KIRKPATRICK

2. Experience with a glucose oxidase method timating glucose in blood and cerebrospinal J. E. Middleton (St. Thomas's Hosp. Med. London, England). *Brit. Med. J.*, 1959, **i**, 26.—The enzyme method of Middleton and hs (*cf. Anal. Abstr.*, 1958, **5**, 3087) used in el with an older chemical method (MacLean) d non-glucose reducing substances to vary 0 to 81 mg per 100 ml (mean 35) in normal liabetic blood. Glucose tolerance can be ed satisfactorily on capillary blood by the e method, but results with the older sugar d are erratic. Glucose fasting levels in l capillary blood range from 46 to 94 mg 0 ml (mean 69), and the normal renal thres- s approx. 160 mg per 100 ml. In normal ospinal fluid, glucose concn. varies from 50 mg per 100 ml (mean 63), and non-glucose ng substances average 19 mg per 100 ml.

H. F. W. KIRKPATRICK

3. The behaviour and characterisation of D- ose in the routine examination of urine. G. (Inst. f. Pharm. Chem. u. Lebensmittelchem., Univ., Marburg/Lahn). *Dtsch. ApothZtg*, 1959, **99** (16), 371–373.—A review is given of the behaviour of galactose in the usual tests for sugars.

A. R. ROGERS

4884. **A sensitive and stereospecific enzymic assay for xylulose.** J. Hickman and G. Ashwell (Nat. Inst. of Arthritis and Metabol. Diseases, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1959, **234** (4), 758–761.—D- and L-xylulose are determined alone, or when mixed, by two enzymes (triphosphopyridine nucleotide-xylitol (L-xylulose) dehydrogenase and diphosphopyridine nucleotide-xylitol (D-xylulose) dehydrogenase which are prepared from guinea-pig liver acetone powder. In the presence of the appropriate reduced pyridine nucleotide the specific dehydrogenase reduces the xylulose to xylitol, and the rate of oxidation of the reduced nucleotide is determined spectrophotometrically at 340 mμ. The triphosphopyridine nucleotide-linked enzyme is specific for L-xylulose, but the other dehydrogenase reacts equally well with D-xylulose and L-erythrulose. J. N. ASHLEY

4885. **Investigation of the fucose content of serum, and of protein fractions. I. Method of determination and normal serum levels.** M. Jacubeit, P. Brünger and M. Knedel (Med. Clinic, Univ. of Munich). *Klin. Wochschr.*, 1959, **37** (8), 460–464.—Serum fucose is estimated by the method of Dische and Shettles for methylpentoses (*J. Biol. Chem.*, 1948, **175**, 595). Three aliquots of 0·05 ml to 0·10 ml of serum are required for each determination—a test sample, an "internal standard" and a serum blank. The serum is deproteinised by the addition of 5 ml of 96% ethanol, and after being set aside for 20 min. the soln. are centrifuged, and the supernatant liquid is discarded; 1 ml of 0·1 *N* NaOH is added to each tube, and to a reagent blank tube, but not to the "internal standard". This contains 0·9 ml of 0·1 *N* NaOH and 0·1 ml of a standard fucose soln. (10 mg per 100 ml). All the tubes are cooled in ice-water, and after the addition of 4·5 ml of 27·8 *N* H_2SO_4 the contents are well mixed and re-cooled. The tubes are then heated in a boiling-water bath for 10 min. and, after cooling under running water, 0·1 ml of a 3% aq. soln. of cysteine hydrochloride is added to each tube except that containing the serum blank, to which 0·1 ml of water is added. After a thorough shaking the colour is allowed to develop by standing the tubes in a bath of running water for 5 hr., in the dark. The soln. are then given a final mixing and all the air bubbles are removed before measuring the colours photometrically at 396 and 430 mμ, with 0·5-cm cells and reading against the reagent blank. The true extinction coeff. is obtained by taking the difference of these two values. The various factors affecting the determination are discussed. Normal values for serum are 8·31 $\pm$ 2·236 mg per 100 ml, or 0·10 to 0·13% in terms of total serum protein, for the 20 subjects studied.

D. B. PALMER

4886. **Quantitative chromatography of carbohydrate constituents of glycoproteins.** J. Montreuil and N. Scheppler (Lab. de Chim. Biol., Fac. de Méd. et Pharm., Lille). *Bull. Soc. Chim. Biol.*, 1959, **41** (1), 13–27.—The amount of carbohydrate determined by hydrolysis and paper chromatography is less by 20 to 50% than that determined by direct colorimetry with orcinol on the pure mucoprotein. Studies on the kinetics of acid hydrolysis of polysaccharides both pure and in

the presence of protein, and combined as muco-protein, show that treatment with 1 *N* HCl for 1 to 4 hr. effects complete hydrolysis of glyco-protein; H_2SO_4 alters the kinetics of the hydrolysis and favours the condensation of sugars with liberated amino acids. Either acid (1 *N* or 2 *N*) is suitable for the hydrolysis of pure polysaccharides which do not contain ketoses. Pentoses are particularly labile. In view of the labile character of certain types of glycosidic bonds, a preliminary systematic investigation of the hydrolysis kinetics for a particular polysaccharide or glycoprotein is recommended. A procedure for the ion-exchange purification and paper-chromatographic analysis of the hydrolysates that gives good recoveries of sugars is described. D. W. Moss

4887. **Separation and quantitative determination by paper chromatography of protein-bound carbohydrates.** R. Bourrillon and J. Michon (Lab. Biochim., Fac. de Méd., 45, rue des Saint-Pères, Paris). *Bull. Soc. Chim. Biol.*, 1959, **41** (2–3), 267–276.—A study of the conditions necessary to obtain optimum separation of protein-bound carbohydrates is presented. By using different chromatographic papers in conjunction with several neutral, acid and alkaline solvents it is shown that S. & S. 2043b acid-washed paper, with any of the following solvents, gives the best separation—*n*-butanol - pyridine - water (5:3:2), *n*-butanol - pyridine - 0·1 *N* HCl (5:3:2) (**I**), *iso*amyl alcohol - pyridine - water (4:4:2) or *iso*amyl alcohol - pyridine - 0·1 *N* HCl (4:4:2) (**II**). The chromatograms are developed by descending chromatography for 16 to 20 hr. at 22° to 23° and, after drying the papers at 100° for 5 min. or at 80° for 30 min., the marker strips are sprayed with a soln. of aniline oxalate. The areas containing the separated sugars are cut out, eluted overnight with double-distilled water at 4°, and aliquots of the resulting soln. are analysed by the methods of Elson and Morgan (*Biochem. J.*, **1933**, **27**, 1824) for osamines, and of Somogyi and Nelson (*J. Biol. Chem.*, 1945, **160**, 61; 1944, **153**, 375) for hexoses and fucose. By using either **I** or **II**, fucose, rhamnose, mannose, glucose, galactose, glucosamine, galactosamine, sialic acid and glucuronic acid can be separated with recoveries of 95 to 103% at levels of 50 to 150 μg of hexoses, 25 to 100 μg of hexosamines, and 20 to 30 μg of fucose. The R_F values of the various compounds are given for the systems used, and the advantages of the method, in particular the separation of glucosamine and galactosamine, are discussed.
D. B. Palmer

4888. **Methods for the isolation of glycolytic intermediates by column chromatography with ion-exchange resins. [Assay of carbohydrate phosphates.]** G. R. Bartlett (Scripps Clinic and Res. Foundation, La Jolla, Calif., U.S.A.). *J. Biol. Chem.*, 1959, **234** (3), 459–465.—To improve the assay of carbohydrate phosphates in eluates from column chromatography, modifications are described and additional information given for the anthrone, carbazole, cysteine - H_2SO_4 and periodate methods of determination. J. N. Ashley

4889. **Studies on materials containing uronic acid. I. An apparatus for routine semi-micro estimations of uronic acid content.** D. M. W. Anderson (Dept. of Chem., Univ., Edinburgh, Scotland). *Talanta*, 1959, **2** (1), 73–78.—A simplified apparatus is described and illustrated for use with the Lefèvre and Tollens decarboxylation reaction. The sample (sufficient to liberate 5 mg of CO_2) is heated under reflux with 35 19% HCl for 2·5 hr. and the CO_2 evolved is re by a stream of N (15 ml per min.), absorb 0·05 *N* $Ba(OH)_2$ and determined by back-tit with 0·05 *N* HCl. Results are quoted fo analysis of glucuronic acid lactone, galact acid monohydrate and Hakea gum. W. T. Car

4890. **Formation of 2-oxo-3-deoxyheptonic in extracts of *Escherichia coli* B. I. Identifi [and determination].** A. Weissbach and J. H (Nat. Inst. of Arthritis and Metabol. Dis Bethesda, Md., U.S.A.). *J. Biol. Chem.*, **234** (4), 705–709.—A very sensitive and s method is described for the detection and mination of sugar acids that contain a 2- deoxy grouping. It depends on the oxidat the deoxy compound with HIO_4 and react the product with thiobarbituric acid to g chromogen with max. absorption at 548 mμ. compound which gives formylpyruvic acid oxidised by HIO_4 will respond to the test.
J. N. Ash

4891. **Colorimetric assay methods for fre phosphorylated glyceric acids.** G. R. Ba (Scripps Clinic and Res. Foundation, La Calif., U.S.A.). *J. Biol. Chem.*, 1959, **23** 469–471.—Spectra are given for the co products formed when glyceric acid, monoph glyceric acid and 2:3-diphosphoglyceric aci heated with 1:3- or 2:7-dihydroxynaphthal chromotropic acid in H_2SO_4 at 100° for 30 Chromotropic acid is the best reagent for the of such compounds and the intensity of the col 695 mμ is proportional to the concn.
J. N. Ash

4892. **Isolation of carotenoids, coumestrol, c genic acid and antibiotics. Application of co current distribution.** C. R. Thompson, A. L and E. M. Bickoff (W. Regional Res. Lab., A Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 841.—The application of the countercurrent bution technique to biologically active mate reviewed briefly and examples showing its us given. A. R. Roc

4893. **Photometric determination of flavo** B. Borkowski and S. Czyszewska. *Biul. Roślin Leczniczych*, 1958, **4** (4), 340–355.— methods for the quant. determination of flav have been described and compared, using and robinin as standard samples. That d Horák was the most sensitive and simple, an found to be useful as a micro-method, its disadvantage being a low stability of the co complex. *Determination of rutin*—Extract of dried, powdered sample (or 2·5 g of fresh sa in a Soxhlet apparatus with methanol - py (20:1) for 90 min. (for a fresh sample do n pyridine). Dilute the extract to 25 ml an 100-μl aliquots on Whatman No. 4 paper to with standard spots of quercetin soln. corresp to 1 to 5 μg of quercetin. Chromatograph descending technique with 60% acetic ac 2 to 3 hr. Determine the quercetin by com the fluorescence with standards under u.v. Cut out the coloured spot due to rutin and e for 30 min. with 5 ml of 0·5% aq. NH_3 soln frequent shaking. Add 1 ml of diazotised sulp acid and shake. After 2 min. add 2 ml of and after 15 min. measure the extinction in a cell with an S42 filter against a blank. W. Ro

4. **Separation of antibodies by starch zone ophoresis.** P. Stelos and W. H. Taliaferro . of Microbiol., Univ. of Chicago, Ill., U.S.A.). *Chem.*, 1959, **31** (5), 845–848.—Two haemo-, of mol. wt. ≃ 160,000 and 1,000,000, ctively, have been separated from rabbit era against sheep red cells by starch zone ophoresis in a barbiturate buffer of pH 8·6 ionic strength 0·1. The antibody recovery d from 70 to 90%. A. R. ROGERS

95. **Phosphorus assay in column chromatoy.** G. R. Bartlett (Scripps Clinic and Res. dation, La Jolla, Calif., U.S.A.). *J. Biol.*, 1959, **234** (3), 466–468.—A modification of Fiske and Subbarow method is described. It sed on the large increase in light absorption 30 mμ which is produced when the reaction ure used in the determination is heated with *N* H_2SO_4 at 150° to 160°. The colour pro-l is stable at room temp. for at least 24 hr., he amount of P is ascertained from a standard . The method (a modification for ultra-micro is also described) has greatly increased tivity and reliability, and is very useful for ple determinations of total P on eluates from natographic columns. J. N. ASHLEY

96. **Colorimetric estimation of serum fatty s.** H. L. Rosenthal, M. L. Pfluke and J. rami (Rochester Gen. Hosp., N.Y., U.S.A.). *Chim. Acta*, 1959, **4** (3), 329–333.—The od of Stern and Shapiro (*J. Clin. Path.*, 1953, 8) is modified and cryst. cholesteryl acetate (**I**) sed as standard. *Procedure*—To 15 ml of r's reagent add 0·5 ml of serum, with shaking, to boiling on a steam bath, cool, and dilute 5 ml with solvent. Prepare a stock standard by dissolving 215 mg of dry **I** in Bloor's nt and dilute 20 ml of this soln. to 100 ml solvent to obtain the working soln. containing mole per ml. To 5 ml each of test filtrate, ing standard and solvent (blank) add with ng 1 ml of 2 *M* hydroxyammonium chloride l ml of 3·38 *N* NaOH, and allow to stand at temp. for 20 to 30 min. Add with mixing of 3·8 *N* HCl and 1 ml of 10% $FeCl_3.6H_2O$ l *N* HCl. After 10 min. read the extinctions 0 mμ. H. F. W. KIRKPATRICK

97. **The DDT content of human fat.** H. r-Bode (Pharmacol. Inst., Univ., Bonn). *w. Chem.*, 1959, **71** (5), 188–189.—The method attson *et al.* (*Anal. Chem.*, 1953, **25**, 1065) was amined. The Davidow column, as used in original procedure, left some impurities in the which reacted similarly to DDT; by using lguhr - H_2SO_4 - Al_2O_3 - Na_2CO_3 as packing, ad of kieselguhr - H_2SO_4 alone, this error eliminated. The highest value for DDT plus [1:1-dichloro-2:2-di-(*p*-chlorophenyl)ethyl-found in 21 samples was 10 p.p.m. Some of the tical results were confirmed by biological riments. A. G. PETO

98. **Fluorimetric method for the differential ation of 3-O-methylated derivatives of adrenand noradrenaline (metanephrine and normetarine).** A. Bertler, A. Carlsson and E. ngren (Dept. of Pharmacol., Univ. of Lund, len). *Clin. Chim. Acta*, 1959, **4** (3), 456–457 English).—The oxidation of these derivatives H 7·2 followed by alkaline treatment yields ounds with activation and fluorescence spectra identical with those obtained from the parent amines. Oxidation at pH 5, however, gives no fluorescence with the methylated compounds, and this can be used to differentiate these derivatives. The following procedure is proposed for the differential determination of metanephrine (**I**) and normetanephrine (**II**). Adjust the pH of the sample to ≃7·2 and to 1 to 3 ml add 0·5 ml of 0·1 *M* phosphate buffer (pH 7·2), H_2O to a vol. of 3·8 ml, and 0·05 ml of 0·02 *N* iodine. After 5 min. add 0·5 ml of 4·5 *N* NaOH containing 0·2% of ascorbic acid. After 5 min. add 0·6 ml of 5 *N* acetic acid and read the fluorescence in a spectrophotofluorimeter as follows—**I**, activation 420 mμ, fluorescence 530 mμ; and **II**, activation 395 mμ, fluorescence 515 mμ. H. F. W. KIRKPATRICK

4899. **3-Hydroxyanthranilic acid metabolism. VII. Mechanism of formation of quinolinic acid.** [**Determination of quinolinic acid.**] S. W. Moline, H. C. Walker and B. S. Schweigert (Div. of Biochem. and Nutr., Amer. Meat Inst. Foundation, Chicago, Ill., U.S.A.). *J. Biol. Chem.*, 1959, **234** (4), 880–883.—Quinolinic acid, after purification on a Dowex-1 anion-exchange column and elution with 0·1 *M* $CaCl_2$, is determined spectrophotometrically at 268 mμ using a predetermined mol. extinction coeff. of 3610. J. N. ASHLEY

4900. **Estimation of α-amino nitrogen by the method of Stanek and Pavlas.** R. Carolan. *Int. Sugar J.*, 1959, **61** (2), 44–45.—Turbidity in the soln. for colorimetry prepared according to the original method (Stanek and Pavlas, *Z. Zuckerind. Czech. Rep.*, 1935, **59**, 129) is avoided by the use of a modified reagent. Dissolve 250 g of cryst. Na acetate in about 450 ml of warm H_2O, and to the cooled soln. add 10 g of copper nitrate dissolved in 200 ml of H_2O. Add 50% acetic acid in 2-ml portions with constant stirring until the pH is between 6·1 and 6·3. Make up to 1 litre, allow to stand overnight and filter. Asparagine standards are used (Stehlík and Cerny, *Ibid.*, 1934, **59**, 284). The blue colour is measured at 650 mμ against a reagent blank. H. F. W. KIRKPATRICK

4901. **Continuous amino-acid separation on an ion-exchange resin by gradient elution with buffer solution.** H. Stegemann (Med. Forschungsanst. d. Max-Planck-Gesellsch., Silikoselabor, Göttingen, Germany). *Naturwissenschaften*, 1959, **46** (3), 110.—Dowex 50-X8, 200 to 400 mesh, prepared in OH form, is used for the column (160 cm × 9 to 10 mm), which is maintained at 40°. Citrate buffer (pH 3·30 ± 0·02) is used for elution, the eluate being collected in 1- or 2-ml fractions and analysed with ninhydrin. All the amino acids are determined separately by this technique.
H. F. W. KIRKPATRICK

4902. **Polychromatic technique for the identification of amino acids on paper chromatograms.** E. D. Moffat and R. I. Lytle (Div. of Biochem., Naval Medical Res. Unit. No. 4, U.S. Naval Training Center, Great Lakes, Ill.). *Anal. Chem.*, 1959, **31** (5), 926–928.—Apply the hydrochlorides of the amino acids to a strip of Whatman No. 1 filter-paper, develop with *n*-butanol - acetic acid - H_2O (4:1:5) for 6 or 7 hr., and dry the strip at 105° for 5 min. Spray with a 50:3 mixture of a 0·2% soln. of ninhydrin in ethanol - acetic acid - 2:4:6-collidine (25:5:1) and a 1% soln. of $Cu(NO_3)_2.3H_2O$ in ethanol, dry the chromatogram at 105° for 1·5 or 2 min., and examine within 15 min. Twenty

common naturally occurring amino acids in quantities $> 1{\cdot}2$ μg are completely differentiated by their individual colours and R_F values. A. R. Rogers

4903. **Paper chromatography of amino acids and other organic compounds in selected solvents.** C. H. Edwards, E. L. Gadsden, L. P. Carter and G. A. Edwards (Dept. of Home Economics and Chem., Agric. and Tech. Coll. of N. Carolina, Greensboro, U.S.A.). *J. Chromatography*, 1959, **2** (2), 188–198.—R_F values are given for 70 organic compounds in phenol - water (**I**) and butanol - propionic acid - water (**II**) systems and also for selected compounds in ethanol - acetic acid, butanol - ethanol - water, butanol - acetic acid - water, and ethanol. The influence of variations in temperature, the presence and absence of buffers and the presence of other compounds in the applied solution are reported. The oxidation of methionine to methionine S-oxide on the paper with **I** and **II** is discussed.
W. T. Carter

4904. **Chromatography of neutral amino acids on columns of cellulose powder.** A. Iachan and J. C. Perrone (Nat. Technol. Inst., Rio de Janeiro, Brazil). *J. Chromatography*, 1959, **2** (2), 213–215.—The effect of the solvent composition on the separation of amino acids on columns of cellulose powder is demonstrated by using ethanol containing 5, 15 or 25% of water. It is concluded that the best separations are obtained by using gradient elution.
W. T. Carter

4905. **Properties of ethyl esters of certain amino acids as related to artefacts in paper chromatography.** J. E. DeVay, A. R. Weinhold and G. Zweig (Univ. of California, Davis, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 815–817.—L-Glutamic acid, L-aspartic acid, β-alanine, and γ-aminobutyric acid (but not other common amino acids), dissolved in 70% ethanol at pH < 3, form appreciable amounts of their (mono)ethyl esters within 36 hr., hence giving extra spots of higher R_F value on chromatography with phenol - water (R_F 0·88, 0·80, 0·83 and 0·80, respectively). Esterification did not occur in 70% ethanol at pH > 3, or in 10% *iso*-propyl alcohol. The chromatography with phenol - water of unesterified glutamic or aspartic acid, at various pH values, gives double spots. Paper electrophoresis confirmed that these were due to dissociation into anions and cations. The changes are reduced to a minimum, or entirely prevented, by adjusting extracts of protein hydrolysates or plant materials to pH 5 after extraction.
D. G. Moss

4906. **The compatibility of profile angles in the identification of substances. Application to the identification of asparagine.** A. C. Shead (Dept. of Chem., Univ. of Oklahoma, Norman, U.S.A.). *Mikrochim. Acta*, 1959, (3), 432–437 (in English).—A critical comparison of data on the angular profile values of asparagine which have been collected over half a century tends to confirm that these values are simply related geometrically and crystallographically. In the absence of distinctive chemical tests and the possibility of using the usual physical constants such as melting- and boiling-points, and the presence of water of crystallisation, one profile-angle measurement can serve for the calculation of all other compatible angles on related crystal forms. This gives promise for the solution of the problem of identification. D. F. Phillips

4907. **Aldehyde oxidation. III. Succi hydic acid dehydrogenase. [Determination aminobutyrate and α-oxoglutarate.]** W. B. J and E. M. Scott (Nat. Inst. of Arthritis and Me Diseases, Bethesda, Md., U.S.A.). *J. Biol.* 1959, **234** (4), 937–940.—Simple and specific are described for $1{\cdot}5 \times 10^{-5}$ to $1{\cdot}5 \times 10^{-4}$ aminobutyrate and α-oxoglutarate. They d on the use of γ-aminobutyric - glutamic aminase coupled with succinaldehydic acid deh genase and the amount of each substrate is mined from the increase in extinction at 3 due to the formation of reduced triphosphopy nucleotide. The rate of reduction of the ox form is linear with respect to enzyme concn.
J. N. Ashi

4908. **γ-Amino-*n*-butyric, aspartic, glutami pyrrolidonecarboxylic acids: their determinatio occurrence in grass during conservation.** Macpherson and J. S. Slater (Chem. Dept., Bldgs., Edinburgh Univ., Scotland). *Biochem* 1959, **71** (4), 654–660.—Glutamic and aspartic are determined in hot-water extracts of gras silage by adsorption on a short column of De-A FF in the acetate form, elution with dil. acetic and assay in the eluate by ninhydrin colorin according to the method of Hirs *et al.* (*J. Chem. Soc.*, 1954, **76**, 6063). Pyrrolidonecarbo acid is determined after hydrolysis to glutamic It is eluted from the column with 5% (v/v) f acid soln. and after evaporation of the eluat residue is heated with 6 *N* HCl to conver cyclic acid into glutamic acid. For the mination of γ-aminobutyric acid the samp applied to a column of Zeo-Karb 225 in the py form. The acid is eluted with 0·2% (v/v) py soln. and determined by the ninhydrin colorin method. J. N. Ashi

4909. **Colorimetric determination of urea special reference to the arginine determin** Yukiaki Nadai (Dept. of Med. Chem., Fac. of Kyoto Univ.). *Acta Med. Univ. Kioto*, 195 (1), 1–5 (in English).—To determine the arg activity of mouse liver and kidney homoge mix the enzyme soln. (1 ml) with 0·05 *M* arg of pH 6 (1 ml), *M* barbitone buffer of p (1 ml) and 0·01 *M* $MnSO_4$ (1 ml) and inc under toluene at 37°. Add 10% trichloro acid soln. (6 ml), set aside for 10 min. and To 5 ml of filtrate, containing 0·15 to 1 of urea, add a mixture of 2% *p*-dimethyla benzaldehyde in 95% ethanol - conc. HCl (2 ml), set aside at room temp. for 15 min. measure the extinction at 450 mμ in a 3-cm
A. R. Rog

4910. **Identification and determination o hydroxyindol-3-ylacetic acid.** H. Wachsmuth L. van Koeckhoven (Hôp. Stuivenberg, An Belgium). *Clin. Chim. Acta*, 1959, **4** (3), 443.—Extraction and chromatography are ca out by the method of Udenfriend *et al.* (*J. Chem.*, 1955, **216**, 499). Reactions with F $K_3Fe(CN)_6$, vanillin, 1:4-naphthaquinone xanthhydrol are described.
H. F. W. Kirkpatri

4911. **Quantitative spectrophotometric deter tion of 5-hydroxytryptamine and 5-hydroxyi 3-ylacetic acid.** A. Carcasona, J. Cervós-Na and F. Unterharnscheidt (Inst. f. Neuropath Univ., Bonn, Germany). *Klin. Wochschr.*, **37** (5), 253–254.—A modification of the meth

end *et al.* (*cf. Anal. Abstr.*, 1956, **2**, 1449) osed. To the acid soln. (1 ml) freed from ng substances add 0·1% ethanolic 1-nitroso- hol soln. (0·5 ml) and 0·0139 *M* HNO_2 warm to 50° to 60° to speed the reaction, act the excess of reagent with 1:2-dichloro- Measure the violet colour at 540 mμ a blank taken through the same process. a standard curve with 5-hydroxytrypt- oln. containing 5 to 80 μg per ml; 5-hydroxy- ylacetic acid is determined similarly, the of reagent being extracted with ethyl (2 × 5 ml). H. F. W. KIRKPATRICK

Determination in urine of some metabolites ophan—kynurenine, anthranilic acid and 3- anthranilic acid—and reference to the e of o-aminophenol in urine. S. L. Tompsett f Clin. Chem., Univ. of Edinburgh, Scotland). *im. Acta*, 1959, **4** (3), 411–419.—Kynurenine ated by distillation and determined by the ation and coupling procedure of Bratton rshall. *o*-Hydroxyamines are separated by ion with diethyl ether and determined with hloro-*p*-benzoquinone-4-chlorimine. H. F. W. KIRKPATRICK

Protein determination for large numbers of . G. L. Miller (Pioneering Res. Div., rmaster Res. and Engng Center, Natick, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 964.— ocedure of Lowry *et al.* (*J. Biol. Chem.*, 1951, 5) has been modified. The reproducibility results is about the same as in the original t the time taken per analysis is substantially l. *Procedure*—Add 1 ml of a mixture (10:1) % soln. of Na_2CO_3 in 0·5 *N* NaOH with a oln. of $CuSO_4$ in 1% K tartrate soln. to ple of protein soln. (1 ml), and after 10 min. luted Folin phenol reagent (1:11) (3 ml) fficient force to ensure mixing. Heat the 50° for 10 min., then cool, and measure the A. R. ROGERS

Stoicheiometry in the estimation of di- e in intact proteins using mercuric chloride. Leach (C.S.I.R.O., Parkville, Melbourne, a, Australia). *Biochim. Biophys. Acta*, 1959, 264–266.—The method is based on the n of soluble proteins with $HgCl_2$ soln. at n the presence of excess of sulphite and of 3 *M*). The end-point is detected ampero- lly with a dropping mercury electrode. isulphide and mercapto groups react, and the requires correction for thiols, which are ined separately by a polarographic method. oluble proteins (*e.g.*, wool), excess of $HgCl_2$ d and complete reaction is obtained in the e of 8 *M* urea in 18 hr. The thiol content is lculated by the described method from the d initial, final and blank diffusion currents) V *vs.* the S.C.E. W. H. C. SHAW

Rapid method for the determination of rotein in serum and plasma with Amido 10B. A. Basso and G. Batacchi (Ist. di Chirurg. Gen., Florence Univ.). *Biochim.* 1959, **6** (2), 65–73.—The staining - elution ue with Amido black 10B (C.I. Acid Black 1) r the quant. determination of protein frac- n paper electrophoresis is modified for the letermination of total protein by applying single spot of serum or plasma on paper. tinction of the eluate at 620 mμ is converted to protein content by means of a standard graph similarly prepared from human serum which has been analysed by Kjeldahl's method. E. C. APLING

4916. **Interaction between dyes and serum proteins in paper electrophoresis. Some quantitative problems.** V. Scardi and V. Bonavita (Inst. of Human Physiol., Univ. of Naples, Italy). *Clin. Chim. Acta*, 1959, **4** (3), 322–328.—The different dye-binding capacities of the albumin and globulins of serum is confirmed in experiments with dyes commonly used in paper electrophoresis. Quant. evaluation is better carried out by determining the albumin and total globulins by chemical means (*e.g.*, with biuret), and limiting the calculation of the pattern to the value of the globulin fractions expressed as percentages of the total globulin content. H. F. W. KIRKPATRICK

4917. **Investigation of the correlation between the results of electrophoresis of serum proteins on paper, on starch gel and by immuno-electrophoresis.** J. Moretti, G. Boussier, M.-P. Hugou and L. Hartmann (Lab. de Biochim., Fac. de Méd., Paris). *Bull. Soc. Chim. Biol.*, 1959, **41** (1), 79–87.—Several pathological sera have been examined by each of the three techniques. Immuno-electrophoresis gives 19 lines of precipitation, 11 of which have been identified; 18 bands are detected by starch-gel electrophoresis. The correlation of these with the 5 zones seen on paper electropherograms has been established. With immuno-electrophoresis the resolution is rather better than on starch gel. Lipoproteins are not shown up by Amido black 10B (C.I. Acid Black 1) in the latter technique, and only poorly by Sudan black B (C.I. Solvent Black 3), while more components of the α- and β-globulins can be demonstrated by immuno-electrophoresis. In clinical work the choice of technique will possibly be governed by the type of abnormality to be assessed. D. W. Moss

4918. **Rapid methods for estimating some serum proteins and the significance of abnormal results.** G. Discombe (Central Middlesex Hospital, London). *Lancet*, 1959, **i**, 619–621.—Total serum protein is determined by the method of Phillips *et al.* (1945), in which a graded series of $CuSO_4$ soln. of known sp. gr. is used to determine the serum sp. gr. This method is rapid, and accurate to ± 0·18 g per 100 ml. Serum albumin is measured by the method of Bracken and Klotz (*Amer. J. Clin. Path.*, 1953, **23**, 1055), which is based on the protein error of indicators, as determined by the change in extinction at 550 mμ of a soln. of methyl orange in the presence of serum. A standard curve is prepared from human or bovine albumin, and covers a range of 1% to 6%; α-, β- and γ-globulins do not interfere, but haemolysis, turbidity and sera from cases of severe jaundice give erroneously low results. Serum γ-globulin is determined by a modification of the $ZnSO_4$ - turbidity method of Kunkel (*Proc. Soc. Exp. Biol. Med.*, 1947, **66**, 217), the $ZnSO_4$ reagent being stored out of contact with CO_2, and the serum sample being added to the reagent with a teat-pipette. The results are expressed in terms of Kunkel's $BaSO_4$ units. Fibrinogen is determined by the method of Stirland (*Lancet*, 1956, **1**, 672). A mixture of plasma and 1% NaCl soln. is heated for 15 min. at 56°, then cooled, and the extinction measured at 660 mμ. Results are expressed as Kunkel units (1 Kunkel unit ≡ 42·8 mg of fibrinogen per 100 ml). For values of >20 units, less plasma must be used.

Each of these methods can be applied to 20 to 30 sera by one technician in a normal working afternoon. The variations in individual serum-protein values and also abnormal distribution patterns in various diseases are discussed. D. B. PALMER

4919. **Borate buffer in zone electrophoresis.** R. Havez and G. Biserte (Lab. de Chim. Biol., Fac. Mixte de Méd. et de Pharm., Lille, France). *Clin. Chim. Acta*, 1959, **4** (3), 334–339.—The influence of the borate buffer in the method of Smithies (*Biochem. J.*, 1955, **61**, 629) in changing the speed of migration of the serum proteins is attributed to interaction of protein and borate ions. The pre-albumin fractions are identified by immunological techniques. H. F. W. KIRKPATRICK

4920. **Ethanediol and propane-1:2-diol in the pre-staining of lipoproteins for electrophoresis.** H. J. McDonald and L. P. Ribeiro (Grad. Sch. and Stritch Sch. of Med., Loyola Univ., Chicago, Ill., U.S.A.). *Clin. Chim. Acta*, 1959, **4** (3), 458–459.—Prepare a soln. of Sudan black B (C.I. Solvent Black 3) by heating ethanediol or propane-1:2-diol to 100° to 110°, adding 1% of dye, stirring for 5 min. and filtering hot through Whatman No. 2 filter-paper. After cooling to room temp., filter the soln. again. To 0·5 ml of serum add slowly with constant gentle mixing 0·1 ml of dye soln. Set aside at room temp. for 45 min., then centrifuge to remove the excess of dye. Use $\simeq 10$ μl of the treated serum per cm width of Whatman 3MM paper for the electrophoresis. These conditions give quant. results in accordance with the Beer - Lambert law. H. F. W. KIRKPATRICK

4921. **Demonstration of plasma fibrinogen by paper electrophoresis.** G. A. Cheyne [Obstet. Med. Res. Unit (M.R.C.), Univ. of Aberdeen, Scotland]. *Lancet*, 1959, **i**, 763.—Electrophoresis of heparinised plasma is carried out by the usual procedure at 8 mA for 24 hr. The polarity is then reversed and the current increased to 12 mA for 1·5 hr. This method yields good separation of the fibrinogen band from other globulins.

H. F. W. KIRKPATRICK

4922. **Determination of fibrin by the heating method of Schulz.** H. Dittrich, E. Puffer and E. Seifert (I Med. Abt. d. Hanusch-Krankenhauses, Vienna, Austria). *Klin. Wochschr.*, 1959, **37** (8), 465–469.—A critical study is made of the method, and the factors influencing the results are investigated. Experiments with pure fibrinogen soln. show variations in the results with different solvents and different quantities of reagents. Under suitable conditions the method has useful clinical application.

H. F. W. KIRKPATRICK

4923. **Isolation and estimation of the sugar of pyrimidine nucleosides and nucleotides after reduction.** L. Haavaldsen, S. Laland, J. M. McKee and E. Roth (The Univ., Blindern, Oslo, Norway). *Biochim. Biophys. Acta*, 1959, **33** (1), 201–206.—The preliminary reduction of pyrimidine nucleotides and nucleosides with sodium amalgam and water permits the subsequent hydrolysis with 0·1 *N* HCl at 100° for 15 min. to be carried out almost quantitatively with respect to sugar. The orcinol or diphenylamine colorimetric methods may then be applied to the determination of the sugar, which may also be characterised by paper chromatography or electrophoresis. The application of the method to thymidine, deoxycytidine and deoxycytidylic and thymidylic acids is described. W. H. C. SHAW

4924. **Interference of phenols in the fluor determination of nucleotides.** A. M. Maye Temp. Res. Sta., Cambridge, England). *Expe* 1959, **15** (4), 158 (in English).—Phenolic subs present in plant tissues cause an apparent phosphopyridine nucleotides when they are mined by the fluorimetric procedure of Lowr (*J. Biol. Chem.*, 1957, **224**, 1047). Substance as catechol, phloroglucinol and quinol h quenching effect on the fluorescence, whil pounds which fluoresce themselves, such as acid, can cause an apparent reduction fluorescence of diphosphopyridine nucleot strong alkali. It is concluded that this m cannot be applied to any tissue in whic phenolic compounds are likely to occur, esp since the amounts of, and ratios between, phenols in plant tissues are liable to markedly during the development of the pla P. NICH

4925. **Rapid paper-chromatographic micr of free and ester cholesterol of blood.** M. L. R. P. Geyer and H. R. Bolliger (Dept. of Nu Harvard Sch. of Public Health, Boston, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 950– After the extraction of serum by acetone - e (1:1), an aliquot of the filtrate is evapora dryness, and dissolved in $CHCl_3$. An aliqu to 20 μl, containing 15 to 75 μg each of fr esterified cholesterol) is spotted on str Whatman No. 1 paper impregnated with and chromatographed by the ascending tec with 1·5% diethyl ether in *cyclo*hexane mobile phase. Standards of cholestero cholesteryl oleate are run simultaneously. solvent front is allowed to ascend about (20 to 30 min.). After location on a guid with molybdophosphoric acid, the segment taining the free and esterified cholesterol a out, and eluted with $CHCl_3$. Cholesterol is mined by a modification of the method of *et al.* (*J. Lab. Clin. Med.*, 1953, **41**, 486). results for total cholesterol agreed within 2% those by two other methods. D. G. M

4926. **Spectrophotometric determination of r Δ^4-3-oxosteroids in bulk $\Delta^{1:4}$-3-oxosteroids. ential rates of thiosemicarbazone formation.** Forist (Dept. of Phys. and Anal. Chem. Upjohn Co., Kalamazoo, Mich., U.S.A.). *Chem.*, 1959, **31** (5), 913–915.—The met based on the difference in the rates of thi carbazone formation, $\Delta^{1:4}$-3-oxosteroids re only slowly under the conditions given. for 12 mixtures containing $< 6\%$ of Δ^4- steroid showed a mean deviation of $\pm$ absolute. *Procedure*—Mix a soln. of the (0·5 mg) in 95% ethanol (5 ml), previously b to 25°, with 0·1 *M* thiosemicarbazide hydroc (1 ml), also at 25°; set aside at 25° for 1 min., then measure the extinction at 302 mμ a a reagent blank. The extinction is a linear fu of the percentage of Δ^4-3-oxosteroid, but for mum accuracy the calibration curve sho checked at the same time as the analysis.

A. R. Ro

See also Abstracts—**4758**, Determination o biological media. **4934**, Lipids in tobacco. Determination of adrenaline. **4956**, Determi of cystine. **4968**, HCN in blood and urine. Artefacts in paper chromatography of amino **5030**, Chromatography of amino acids. **5053**, mination of enzyme activity.

Pharmaceutical analysis

Application of paper chromatography to tection of alkaloids in toxicological food s. J. Kolankiewicz and M. Nikonorow of Food Res., Inst. of Hygiene, Warsaw). olon. Pharm., 1959, **16** (2), 115–122.—The hromatographic method for the detection loids, *e.g.*, codeine, veratrine, quinine and ne, in several foods (a 100-g sample) is ed. For the preliminary extraction of the ds add to the sample 300 ml of ethanol d to litmus paper with HCl. Digest on a ath at 40° for 24 hr., filter and retain the . Add 100 ml of acidified ethanol to the , digest for another 12 hr. and filter again. ne the filtrates and remove the alcohol by ating on a water bath at 40°. Add 30 ml of and extract with diethyl ether (5 × 50 ml). aOH soln. to the aq. layer till it is alkaline to and extract with diethyl ether (5 × 50 ml). rate the ether extract to 50 ml, transfer it separating-funnel and wash with 2 *N* HCl 20 ml). To the combined acid washings add till they are alkaline to litmus and extract iethyl ether (3 × 50 ml). Evaporate the completely and dissolve the extract in 5 ml nol. Retain this alcoholic soln. for chroma- hy by the descending technique on Whatman paper. Apply drops of the test and standard o the paper and impregnate its whole area the starting line with a freshly prepared lic soln. of formamide (1:1). Dry the paper n two filter-papers, then at 40° for 30 min. the starting points with the ethanolic soln. of nide (1:1) and after 10 min. transfer the to a glass container containing $CHCl_3$ at 25°. evelopment is complete in 2·5 hr. Dry the atogram at 105° and spray with a soln. of ium iodoplatinate, washing off the excess ater in order to observe the spots on the white round. The following amounts of alkaloids e detected—codeine 0·01 to 0·3 mg, quinine to 0·015 mg, veratrine 0·1 to 0·15 mg and ine 0·05 to 0·075 mg. W. Roubo

3. **Determination of the origin of opium. II. aneous assay of narcotine, thebaine and erine in opium by infra-red spectroscopy.** Bakre, Z. Karaata, J. C. Bartlet and C. G. lo (Food and Drug Directorate, Dept. of Iealth and Welfare, Ottawa, Ontario, Canada). arm. Pharmacol., 1959, **11** (4), 234–243.— ate the sample (4·5 g as 20 to 40-mesh r) for 10 to 20 min. with glacial acetic acid), slowly add H_2O (25 ml) with continuous g and filter through paper. Extract a 10-ml t of the filtrate with $CHCl_3$ (4 × 10 ml). each extract successively with H_2O (10 ml), sodium bisulphite in 30% aq. NaOH soln.), H_2O (10 ml) and H_2O (10 ml). Filter the ned $CHCl_3$ soln. through cotton wool and rate. Dry the residue in a desiccator, mix it nhydrous CCl_4, filter through sintered glass ilute the filtrate to 25 ml. Examine the i.r. um from 1100 cm^{-1} to 1900 cm^{-1} in a 1-mm n chloride cell. Calculate the percentages of ine, thebaine and papaverine from the extinc- at 1767, 1602 and 1160 cm^{-1}. The results samples of opium were compared with those ed by the United Nations K/34 method a modified method (*cf.* Lee and Farmilo, *Abstr.*, 1959, **6**, 1888). The proposed i.r. d needs only half a day per sample, and the reproducibility is two or three times as good as that of the chemical methods. A. R. Rogers

4929. **Determination of small amounts of hyoscyamine and hyoscine in crude drugs.** J. W. de Bruyn and J. G. van Hall (Inst. v. Vered. van Tuinbouw., Wageningen). *Pharm. Weekbl.*, 1959, **94** (8), 262–269.—The alkaloids are separated by paper chromatography with the solvent *n*-butanol - glacial acetic acid (10:1) saturated with water. The spots are eluted and the alkaloids are determined by the method of Häussler (*Anal. Abstr.*, 1958, **5**, 1662) by measurement of the coloured complex with Tropaeolin OO (C.I. Acid Orange 5). Quantities of 10 to 150 μg can be determined. Hyoscyamine can be determined with methyl orange at pH 8; hyoscine does not react. N. E.

4930. **Assay of strychnine in pharmaceutical preparations of nux vomica.** I. S. Taylor and A. Ifrim (Commonwealth Lab., Melbourne, C.I.). *J. Pharm. Pharmacol.*, 1959, **11** (3), 191.—Render alkaline an aliquot of the sample (containing ≃ 0·5 mg of strychnine) and evaporate to a vol. of ≃ 2 ml. Mix the residue with active alumina (heat alumina at 800° for 6 hr.) (1 g) and transfer to a chromatographic column of active alumina (10 g) packed "wet" with ethanol. Elute with ethanol (50 ml) at a rate of 5 to 10 drops per min. and allow the column to drain. Evaporate the eluate to dryness, dissolve the residue in *N* H_2SO_4 (100 ml) and measure the extinction at 262 mμ (*A*) and 300 mμ (*B*) against *N* H_2SO_4 in 1-cm quartz cells. Then strychnine (%) = (0·318 *A* − 0·460 *B*)/100. A. R. Rogers

4931. **Determination of ephedrine in ephedra by non-aqueous titration.** Yun-Han Ho and Teng-Han Tang. *Acta Pharm. Sinica*, 1959, **7** (3), 119–121.—The simplified titrimetric method in non-aqueous soln. (*cf.* Chatten and Pernarowski, *Anal. Abstr.*, 1954, **1**, 1626) is suitable for the determination of ephedrine in ephedra and is applicable to ephedrine tablets and ampoules. *Procedure*—Weigh out finely powdered ephedra (10 g) into a 500-ml stoppered flask, add 1 *N* HCl (40 ml) and heat on a water bath for 1 hr. After cooling, neutralise with 6 *N* NaOH and add $CHCl_3$ (100 ml). Stopper the flask and shake vigorously for 15 min. Add powdered tragacanth (6 g) and shake for a further 5 min. Filter and transfer the $CHCl_3$ filtrate (50 ml ≡ 5 g of sample) to a clean flask. Add 6% mercuric acetate soln. (5 ml) and 0·5% crystal violet indicator (1 drop) and titrate with 0·1 *N* $HClO_4$ in glacial acetic acid until the colour changes to green. The results for 3 samples by the method described are in agreement with those obtained by the method of the Chinese Pharmacopoeia, 1953. S. H. Yuen

4932. **Stability of rescinnamine in solution.** The Pharmaceutical Society of Great Britain and The Society for Analytical Chemistry (Anal. Methods Committee, 14 Belgrave Sq., London). *J. Pharm. Pharmacol.*, 1959, **11** (4), 211–215.—Results of an investigation by a Joint Committee on Methods of Assay of Crude Drugs are presented. The u.v. absorption characteristics of rescinnamine change when soln. in various solvents are exposed to daylight, but there is no detectable alteration in the pharmacological properties. Soln. of trimethoxycinnamic acid, but not of trimethoxybenzoic acid, show similar changes. It appears that the method of assay of rauwolfia proposed by Carol *et al.* (*cf. Anal. Abstr.*, 1956, **3**, 2847) cannot be regarded as trustworthy. A. R. Rogers

4933. **Photometric micro-determination of lupin alkaloids.** M. Wiewiórowski and J. Skolik (Inst. f. allg. Chem., Ökonom. Hochsch., Poznań). *Roczn. Chem.*, 1959, **33** (2), 461–469 (in German).—The method permits the separation and determination of $\simeq$ 20-μg quantities of lupinine, lupanine, hydroxylupanine, angustifoline and sparteine with an error $< \pm$ 3%. *Procedure*—Moisten the crushed seeds (300 to 400 mg) with 2 *N* KOH (6 drops), mix with kieselguhr (600 mg) and pack into a column for chromatography. Elute with $CHCl_3$ (9 ml) so that the eluate drips on to filter-paper. Allow the $CHCl_3$ to evaporate, acidify the extracted spot with acetone - acetic acid - H_2O (10:1:6) and dry the paper over KOH and P_2O_5 *in vacuo*. Wash the alkaloids on to a paper chromatogram with methanol and develop with H_2O-satd. *n*-butanol. Dry the paper and expose it to iodine vapour to reveal the positions of the separated alkaloids. Cut out the zones and extract each separately with H_2O (3 ml). Add a 0·0002 *M* soln. of bromothymol blue in borate buffer (pH 8·0) (5 ml) to the aq. extract and shake for 5 min. with $CHCl_3$ (10 ml). Mix 5 ml of the $CHCl_3$ layer with 0·002 *N* methanolic KOH (5 ml) and measure the extinction in a Lange colorimeter with an orange filter OG2.

A. R. Rogers

4934. **Estimation of the lipid fractions of tobacco.** D. N. Hellier (Res. Dept., Carreras Ltd., 221 Stanhope St., London). *Chem. & Ind.*, 1959, (8), 260–261.—Absolute ethanol is found to be more effective than light petroleum for the extraction of tobacco when determining tobacco resins. *Procedure*—Dry ground tobacco (30 g) is extracted in a Soxhlet apparatus for 10 hr. with 150 ml of ethanol. The extract is concentrated to 100 ml and 5 ml is streaked on to Whatman No. 3 paper. Water-soluble compounds are removed by descending chromatography with 2% aq. acetic acid. After air-drying, the base-line strip is eluted successively with light petroleum, diethyl ether and ethanol to give "soft resin", "hard resin A" and "hard resin B" fractions, respectively. Analyses for various growing areas are given, and the contents of the various fractions are discussed. E. J. H. Birch

4935. **Micro-determination of adrenaline by titration of the ferrocyanide formed from ferricyanide.** J. A. C. van Pinxteren and M. E. Verloop (Farm. Lab., Rijksuniv., Utrecht, Holland). *Pharm. Weekbl.*, 1959, **94** (6), 169–178.—Adrenaline is quant. oxidised with $K_3Fe(CN)_6$ to adrenochrome, and the resulting ferrocyanide is determined by a dead-stop end-point titration with 0·015 *M* $ZnSO_4$. The results are vitiated by the presence in pharmaceutical preparations of sodium metabisulphite and phenol; these substances are eliminated by passing a current of N through the test sample (2 ml), slightly acidified with dil. H_2SO_4, for 10 min. at 100°. The cooled sample is then neutralised and mixed with aq. 2% $Na_2B_4O_7$ soln. (2 ml); at the resulting pH (7·6 to 9·3), the time for oxidation with $K_3Fe(CN)_6$ (5 ml of 0·05 *M* soln.) is reduced to 5 min. The soln. is slightly acidified before titration. The results show good reproducibility and are $\gtrsim$2% high. P. S. Arup

4936. **Quantitative analysis of senna leaf by chemical methods.** M. Henneberg (Zaklad Anal. i Technol. Leku Roslinnego). *Biul. Inst. Roślin Leczniczych*, 1959, **5** (1), 1–10.—To establish the most suitable method for the quant. determination of the active principle of senna leaf two chemical methods were compared, and after modifi in particular to the method of extraction, hoff's method (*Arzneimittel-Forsch.*, 1951, 1 was considered to have some advantage Kussmaul and Becker's method (*Helv. Chim* 1947, **30**, 59), especially for a series of dete tions, as it is much shorter, simpler and suffi accurate in comparison with pharmaco estimations. On the basis of results obtai chemical and pharmacological methods, it i posed that the minimum content of sennos senna leaf should be 2·2%. W. B. Miasko

4937. **Determination of anthraquinone tives in proprietary medicines. I. Photo determination of frangula emodin in Ch** L. Kraus (State Inst. for Control of Drugs, P *Českosl. Farm.*, 1959, **8** (2), 83–84.—The me based on the classical Borntträger reactio KOH and was applied to Cholagol (a mixt frangula emodin, curcumin, volatile oils, e and olive oil). *Procedure*—Boil the sample (a 1 g $\equiv$ 0·15 mg of emodin) for 20 min. under with NaOH soln. (20%) (20 ml), then heat s for 5 to 8 min. and filter the pink soln. 100-ml flask. Wash the filter with NaOH (20%) (20 ml) into a beaker and warm the f Repeat this procedure three times. Cool th bined filtrates, dilute to vol. with NaOH (20%), mix, and measure the extinction in cell with the use of filter S53. Compare a calibration curve. J. Z

4938. **Polarography of santonin.** Shu-Ha and Tong-Hui Chow. *Acta Pharm. Sinica*, **7** (1), 17–24.—Pure santonin is studied in di buffers. It is found that, below pH 3·5, on wave is obtained, corresponding to a one-el reversible reduction; the $E_{\frac{1}{2}}$ shifts more and towards the negative with increasing pH. range pH 3·5 to 9, two waves are observe first being the same as that found below p and the second is a one-electron irreversible tion. At pH values $>$ 9, the first wave into the second one, resulting in a single corresponding to a two-electron reduction. effects of the mercury height and temp. limiting current show that the latter is dif controlled. For the analysis, a medium con ethanol (25% v/v) and a buffer (boric acid NaOH) (pH 10) are recommended. The height is measured at $-$ 1·7 V *vs.* the Santoninic acid behaves similarly to santoni slightly higher negative $E_{\frac{1}{2}}$, but santoni formed by the vigorous treatment of santoni alkali is polarographically inactive. Fro results given by pure santonin and from a st the extraction of santonin from artemisia, cedure for the rapid analysis of artemisia posed. The sample may be analysed in 90 min. and the results agree with those ob by the gravimetric method. Sci. Abstr. C

4939. **Methods for the determination of t** K. Herrmann and W. Enge (Inst. f. Lan Technol. u. Vorratspflege, Martin Luther Halle-Wittenberg, Germany). *Dtsch. Ap* 1959, **99** (14), 325–328.—Results for tan vegetable drugs obtained by the routine iod method (in which the oxidative value of the is determined before and after the removal tannins) and by the colorimetric tungstoph method are generally in good agreement wit obtained by the standard gravimetric m

obtained by the Cu acetate gravimetric
d are from 7 to 19% low. All the available
ds are subject to possible errors due to the
on of certain non-tannins (chiefly polyhydric
s) in the determinations. Specific methods
be developed for the separate determination
substances. (35 references.) P. S. ARUP

. Titration of alkali salts of organic acids erchloric acid in non-aqueous medium. M. R. Lux and E. Franken (Pharmaceut. Inst., Univ., Germany). *Dtsch. ApothZtg*, 1959, 157–158.—Several alkali and alkaline-earth f organic acids, contained in D.A.B. VI, were ined by titration with 0·1 *N* $HClO_4$ in acetic acid, with Oracet blue B (C.I. Solvent 9) as indicator. The method is similar to or the determination of organic bases (*cf. t al., Anal. Abstr.*, 1959, **6**, 1885).
M. H. SAWISTOWSKA

. Determination of barbiturates in tablets. éguin (Lab. de Pharm. Galénique, Univ., e). *Pharm. Acta Helv.*, 1959, **34** (3), 146–n French).—Grind and extract the powdered e (containing 0·6 to 0·9 g of barbiturate) with . NH_3 (4 × 9 ml). For a sodium barbiturate, 5 *N* aq. NH_3; for calcium cyclobarbitone, use ml portions of 2 *N* aq. NH_3. Centrifuge each t and dilute the combined soln. to 50 ml. y a 25-ml aliquot of the soln. with 5 *N* HCl xtract with $CHCl_3$ (3 × 25 ml). For phenone, use $CHCl_3$ - diethyl ether (4:1) (3 × ; for calcium cyclobarbitone, $CHCl_3$ (4 × . Evaporate the combined extracts, dry the e at 103° to 105° and weigh. If the excipient ns sodium carboxymethylcellulose, extract wdered sample with 95% ethanol, evaporate tract and assay the residue. Remove Carbo-000 by shaking the ammoniacal soln. with - diethyl ether (4:1) before the addition of Benzoic acid and its salts interfere, but be removed. The precision of the method is o 1·5%. A. R. ROGERS

. X-ray diffraction patterns of *p*-nitrobenzyl ives of barbituric acids. J. A. R. Cloutier M. Manson (Dept. of National Health and e, Ottawa, Ontario, Canada). *Appl. Spectro-* 1959, **13** (2), 34–38.—Diffraction data for -nitrobenzyl derivatives of 20 clinically ant barbituric acids are presented.
K. A. PROCTOR

. Coulometric micro-titration of thiopentone n and of rivanol [ethacridine lactate]. K. wski and A. Piotrowska (Dept. of Pharm. Med. Acad., Lódź). *Acta Polon. Pharm.*, **16** (2), 107–109.—The determination of thioe sodium (**I**) and ethacridine lactate (**II**) the help of electrically generated Br is ed. The dead-stop method was used with atinum electrodes in a specially constructed *Determination of* **I**—Add to the anode comnt 3 ml of 30% KBr soln., 3 ml of 10% ln. and 2 ml of the test soln. and make up ml with water. To the cathode compartment veral ml of 10% HCl soln. so that its level ner than that in the anode compartment. on the mixing device in the anode comnt, then the indicating circuit and finally nerating system and measure the time from int every 0·2 sec. Continue the electrolysis e presence of free Br is indicated. Carry blank determination with water instead of the test soln. A current of 8 mA is used. *Determination of* **II**—Add to the anode compartment 5 ml of 20% KBr soln., 5 ml of 15% H_2SO_4 soln. and 2 ml of the test soln. and make up to 25 ml with water. To the cathode compartment add 15% H_2SO_4 soln. till its level is higher than that in the anode compartment and then proceed as for **I**. The amount of each substance is calculated from the equation given. The method permits the determination of from 0·3 to 1·5 mg of **I** with ± 0·7% accuracy, and of 1 to 3 mg of **II** with ± 1% accuracy. It takes only a few minutes, is simple and needs only a small amount of sample.
W. ROUBO

4944. **Spectrophotometric determination of methadone.** M. J. Pro (Alcohol and Tobacco Tax Div. Lab., Int. Rev. Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (1), 117–180.—Methadone is extracted from tablets with water and the extinction of the soln. at 292·5 mμ is read. Alternatively, the sample may be extracted with $CHCl_3$ and the extinction read at 5·86 μ. The procedure for the separation of methadone from interfering substances is described.
A. A. ELDRIDGE

4945. **Micro-method for the estimation of anaesthetics derived from aminobenzoic acid. Procaine hydrochloride and tetracaine [amethocaine] hydrochloride combined in cartridges for dental anaesthesia.** M. E. Auerbach and M. M. Tuckerman (Sterling-Winthrop Res. Inst., Rensselaer, N.Y., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (3), 194–196.—Mix the sample [1 ml containing 20 mg of procaine hydrochloride (**I**) and 1·5 mg of amethocaine hydrochloride (**II**)] with H_2O (20 ml) and 10% Na_2CO_3 soln. (2 ml) and extract with $CHCl_3$ (3 × 25 ml). Wash the combined extracts with H_2O (15 ml), filter through paper, add 7 *N* ethanolic HCl (0·2 ml), evaporate to dryness and dissolve the residue in H_2O (100 ml). Determine the total anaesthetic in a 25-ml aliquot by dilution with 7 *N* H_2SO_4 to 50 ml and measurement of the extinction at 272 mμ. To determine **II** alone, mix a 50-ml aliquot with 7% $NaNO_2$ soln. (1 ml) and add *N* HCl (1 ml). After 5 min., add 10% Na_2CO_3 soln. (2 ml) and 1% Na 8-amino-1-naphthol-2:4-disulphonate soln. (2 ml), set aside for 1 min. and extract with $CHCl_3$ (3 × 25 ml). Wash the combined extracts with H_2O (15 ml), filter through paper, dilute with $CHCl_3$ to 100 ml and measure the extinction at 290 mμ against a blank prepared by similar treatment of a 0·02% soln. of **I** (50 ml).
A. R. ROGERS

4946. **New method for the colorimetric determination of sodium 4-aminosalicylate.** G. Baiulescu (Chem. Res. Centre, Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1959, **6** (3), 433–438.—The method is based on the formation of an orange-coloured complex with uranyl ions which obeys the Beer - Lambert law. A soln. of uranyl nitrate (2·5% in methanol) is used as reagent, and a 0·3% soln. of Na 4-aminosalicylate (**I**) for preparing the standard curve. The maximum absorption is at 420 mμ. A Pulfrich photometer with S42, S43 and S47 filters is used. Under the conditions given, the determination of 0·75 to 6 mg of **I** is not affected by the presence of 1 mg of isoniazid nor by *m*-aminophenol.
H. SHER

4947. **Photometric determination of some derivatives of salicylic acid.** O. Hrdý and J. Černý (State Inst. for Control of Drugs, Prague), *Českosl. Farm.*, 1959, **8** (2), 73–77.—The method is based on the

condensation reaction with *p*-amino-NN-diethylaniline sulphate (**I**) in an oxidising medium of $K_3Fe(CN)_6$. *Procedure*—To the ethanolic (96%) soln. containing, in 5 ml, salicylamide (**II**) (5 to 100 μg), phenyl salicylate (**III**) (10 to 200 μg) or methyl salicylate (**IV**) (10 to 200 μg) add $NaHCO_3$ soln. (5%) (2 ml) for **II**, or 0·05 *M* $Na_2B_4O_7.10H_2O$ - 0·1 *N* NaOH (1:1) (2 ml) for **III**, or 0·05 *M* $Na_2B_4O_7.10H_2O$ - 0·1 *N* NaOH (7:3) (2 ml) for **IV**, followed by a soln. of **I** (0·2% for **II**, or 1% for **III** or **IV**) (1 ml) and $K_3Fe(CN)_6$ soln. (2% for **II**, 10% for **III** or **IV**) (1 ml). Mix and add benzene (10·0 ml) and shake for ≃ 30 sec. Dry the benzene layer with anhyd. Na_2SO_4 and measure the colour in a Pulfrich photometer (filter S61). The method was applied with good results to the determination of **II**, **III** and **IV** in pharmaceuticals. The mechanism of the reaction is discussed. J. Zýka

4948. **Circular paper chromatographic technique for the separation of mixtures of sulpha drugs.** Swarup Narain Tewari and Dharam Narain Tripathi (Clin. Lab., The Mall, Kanpur, India). *Z. anal. Chem.*, 1959, **166** (5), 356–357 (in English).—*Procedure*—One drop of the mixture (dissolved in aq. NH_3) is placed at the centre of a Whatman No. 1 filter-paper (18 cm diam.). The solvent-feeding wick is applied to the centre of the spot on the paper, which is placed on a Petri dish and covered with another dish. Acid (*n*-butanol - acetic acid - water, 5:1:4) and alkaline (*n*-butanol - conc. aq. NH_3, 5:1) solvents are used. After the development the disc is dried, and sprayed with a 1% soln. of *p*-dimethylaminobenzaldehyde in 3% HCl to give orange - yellow circular bands, which are compared with known chromatograms. R_F values for 8 sulphonamides are listed for each solvent.
B. B. Bauminger

4949. **Simultaneous colorimetric determination of chloramphenicol cinnamate and sulphonamides.** G. Machek (Kontroll-Lab. der Biochem. G.m.b.H., Kundl, Tirol). *Sci. Pharm.*, 1959, **27** (1), 24–34.—To determine sulphonamides, diazotise the diluted sample, couple with N-1-naphthylethylenediamine and measure the extinction at 540 mμ. To determine chloramphenicol cinnamate, reduce the diluted sample with alkaline sodium dithionite before application of the colorimetric method and use the coloured soln. from the first determination as the blank. The method has been applied to the assay of syrups and 'chartae' (*cf. Anal. Abstr.*, 1958, **5**, 4282). Results are accurate to within about ± 2%.
A. R. Rogers

4950. **The dead-stop titration. V. Dead-stop titration, derivative polarographic titration, and potentiometric polarography of sulphanilamide derivatives by diazometry with two platinum electrodes.** Takehisa Enoki and Katsuaki Morisaka (Coll. of Pharm., Kitayashita, Minami-kawachi-gun, Osaka). *J. Pharm. Soc. Japan*, 1958, **78** (4), 432–435.—The dead-stop titration of aromatic amines making use of the redox reversible system with HNO_2 was studied with reference to the influence of temp. and the rate of titration. By the use of 0·1 *N* $NaNO_2$ in *N* H_2SO_4 at 0°, the change in current for dead-stop titration with a potential difference of 150 mV and that in potential for derivative polarographic titration (3 μA) are 5 μA and 50 mV, respectively, at the end-point, corresponding to an error of 5% for sulphanilamide and 3% for aniline. By the use of 0·5 *N* $NaNO_2$, the error is decreased to one fifth. Near the end-point, the rate of titration is decreased to 1 ml per min.
K. Saito

4951. **Complexometric determination of derivatives of phenothiazine. I. Determi of chlorpromazine.** L. Przyborowski a Krówczyński (Dept. of Pharm. Chem., Med. Lublin). *Chem. Anal., Warsaw*, 1959, **4** 59–67.—The method consists in pptg. promazine (**I**) from soln. by means of an ex a standard soln. of CdI_2, in the presence of HI. A ppt. of the formula $(C_{17}H_{19}N_2SCl)_2.$ is formed, and the excess of CdI_2 in the filt determined complexometrically, with X orange as indicator. The method is suital determining **I** as the pure substance and in pl ceutical preparations (*e.g.*, tablets and injec it takes only 30 min., and is satisfactorily ac despite a small loss of the determined substan
W. B. Miasko

4952. **Comparison of the methods for the mination of nikethamide.** Heng-Yun Y Duenn Tao (Anal. Dept., First Nat. Pharm. Shanghai). *Acta Pharm. Sinica*, 1959, **7** (3) 125.—The simple rapid method for the dete tion of nikethamide (*cf.* Fritz, *Brit. Ab* 1951, 49) is based on the non-aqueous titra glacial acetic acid with $HClO_4$ soln. in acetic acid, the end-point being detected po metrically, or visually by the use of crystal as indicator. *Procedure*—Weigh 0·2 g of into a 100-ml conical flask and dissolve in acetic acid (5 ml) that has previously been ac to a bluish-green with $HClO_4$, with crystal as indicator. Add 0·5% crystal violet s drops) and titrate with 0·1 *N* $HClO_4$ in acetic acid to a bluish-green end-point. Th posed method gives a standard error of ± 0 and is more accurate than the hydroly refractometric methods. S. H. Y

4953. **Determination of diethylenediamine azine].** J. Erben (State Inst. for Control of Prague). *Českosl. Farm.*, 1959, **8** (1), 18 The method is based on the reaction of pip (**I**) with $HgCl_2$ soln. and on the complexo determination of the excess of reagent aft removal of the ppt. *Procedure*—Dissolve **I** in H_2O (10 ml), add 0·1 *M* $HgCl_2$ (30·0 ml) the mixture slightly, then cool, and filter c wash the ppt. with H_2O. To the filtrate a excess of 0·05 *M* EDTA (disodium salt), th NH_3 - NH_4Cl buffer soln. (pH 10), dilute to and titrate with 0·05 *M* $MgSO_4$, with Erioc black T (C.I. Mordant Black 11) as indicator.
J. Z

4954. **Polarographic behaviour of iodinated contrast agents in buffered media.** P. Kabasa and J. McGlotten (Schering Corp., Bloomfield U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 51 —Ten X-ray contrast agents containing iodin studied polarographically in 50% ethanol at various pH values between 1·3 and 10·5 effects of temp., sample concn. and pH on th form and height were examined. All the pounds tested gave well-defined and reproc polarograms at some pH in the system use the diffusion currents were linear with resp concn. over a fairly wide range. G. P. C

See also Abstracts—**4819**, Determinati formaldehyde and paraformaldehyde. **4821**, mination of formaldehyde in technical hex **4827**, Titration of sulphonamides. **4892**, Is

ıestrol and antibiotics. **4976**, Determination mine. **4978**, Separation of pyridoxine and c acid. **5016**, Determination of halothane ıoroform vapour.

Food

ods and food additives, beverages, edible oils and fats, vitamins.

Simple method for determining the protein of wheat and flour. L. Feinstein and J. R. Biol. Sci. Branch, AMS, U.S. Dept. of Agric., lle, Md., U.S.A.). *Cereal Chem.*, 1959, **36** –193.—A rapid turbidimetric method, which eproducible results, is described. *Procedure*—flour or ground wheat (1·0 g) with 100 ml *N* NaOH for 15 min., and centrifuge the sion at 6000 r.p.m. for ∢ 10 min. Dilute a liquot of the clear supernatant liquid to vith aq. sulphosalicylic acid soln. (3%, w/v), ıoroughly, and immediately measure the ty of a 4-cm layer in a photo-electric colori- with a 540-mμ filter against a blank of salicylic acid soln. Determine the protein erence to a calibration curve. S. C. Jolly

Polarographic determination of cystine in and flour hydrolysates. W. F. Daum, J. E. es and B. S. Miller [Standard Oil Co. (Ind.), sha, Kans., U.S.A.]. *Cereal Chem.*, 1959, **36** 6–185.—The cystine content of flour from erent varieties of wheat determined by this ed polarographic method agreed well with determined by chemical and microbiological ds. *Procedure*—Heat ground wheat or flour with 4·0 *N* HCl (25 ml) for 0·5 hr. at 15 lb re, cool rapidly, dilute the hydrolysate to , and filter. Dilute a 3-ml aliquot of the l hydrolysate to 50 ml with electrolytic n (1·6 × 10^{-3} *M* $CoCl_2$, 0·105 *M* NH_4Cl and *M* aq. NH_3, prepared fresh each day from soln.), and record the polarogram within of mixing. Measure the cystine wave-height e difference in mm between the diffusion t of Co^{2+} (obtained from the electrolytic m with no added cystine) and the minimum catalytic wave due to cystine (Wostmann, 1950, **27**, 391). Prepare a standard curve ding increments (2·0 to 12·0 × 10^{-2} mg) of e to 3-ml aliquots of the filtered hydrolysate ıich the cystine has been destroyed by treat- with 10 ml of 1% H_2O_2 for 17 hr. at 18° °, and decomposing the excess of H_2O_2 by g on a steam bath for 30 min.), diluting to 50 th electrolytic medium, and recording the gram. S. C. Jolly

. Variations in the gross composition of milk ated to the breed of the cow: a review and l evaluation of the literature of the United and Canada. T. V. Armstrong (Ohio State Columbus, U.S.A.). *J. Dairy Sci.*, 1959,), 1–19.—A review is presented, with 53 ıces. W. H. C. Shaw

3. Determination of degradation products of ic acid [in milk]. J. Tobias and E. O. Herreid is Univ., Urbana, U.S.A.). *J. Dairy Sci.*, **42** (3), 428–440.—The method described is on reaction with 2:4-dinitrophenylhydrazine e determination of ascorbic acid, dehydro- ic acid ("total" ascorbic acid) and 2:3-L-gulonic acid (**I**). **I** is then determined separately after treatment with thiourea and H_2S. Results are compared with those obtained by the 2:6-dichlorophenolindophenol method. Small amounts of **I** were found in raw milk (1 to 2 days old), but little or none in dried skim or evaporated milk. The **I** content of pasteurised milk, initially low, increased considerably on storage at 40° F.

W. H. C. Shaw

4959. **Application of the chemical inhibition method for vitamin D to evaporated milk.** S. W. Jones, W. W. Morris and J. B. Wilkie (Div. Nutrition, Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (1), 180–186.—The method used for pharmaceutical preparations (*cf.* Wilkie *et al.*, *Anal. Abstr.*, 1959, **6**, 1538) is modified. Fat is extracted before saponification; for this purpose two extractions with diethyl ether suffice. After saponification it is necessary to employ two columns of MgO - Hyflo Supercel, followed by one of Al_2O_3. In the Al_2O_3 column the carotene is eluted with 1 ml of diethyl ether and then with 6 ml of light petroleum. The $SbCl_3$ reagent is used to give a colour complex, which is measured at 500 mμ, contributory colour produced by vitamin-A decomposition products being allowed for by employing acetic anhydride to inhibit the formation of that due to vitamin D.

A. A. Eldridge

4960. **Analysis of milk for rotenoids.** M. Jacobson, I. Hornstein and R. T. Murphy (Entomol. Res. Div., Agric. Res. Service, U.S. Dept. Agric., Beltsville, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (1), 174–176.—Rotenoids in milk, resulting from the use of sprays containing rotenone on cattle, may be detected by a spectrophotometric or a chromatographic method. The butter fat is extracted with a mixture of methyl cyanide and aq. Na_2SO_4 soln.; the soln. is washed with hexane and then extracted with $CHCl_3$, which is subsequently evaporated, and the residue is dissolved in CCl_4. The soln. is then chromatographed in a specially prepared alumina column, and eluate fractions are examined spectrophotometrically at 270 mμ. Alternatively, the CCl_4 is removed by evaporation and the residue, dissolved in acetone, is spotted on paper. Methanol - benzene - water (5:1:1) is used for development, followed by a $KMnO_4$ spray. The limits of sensitivity are 0·04 and 0·006 p.p.m., respectively, for rotenone and dehydrorotenone. A. A. Eldridge

4961. **Rapid spectrophotometric method for measuring cheese ripening.** D. G. Vakaleris and W. V. Price (Wisconsin Univ., Madison, U.S.A.). *J. Dairy Sci.*, 1959, **42** (2), 264–276.—The method is based on the u.v. absorption of a Na citrate - HCl extract of the sample; the extinction is almost entirely due to water-soluble tyrosine and tryptophan, which are determined by a two-component analysis. Results on 40 samples of Cheddar cheese correlated well with the water-soluble N (Kjeldahl method) and with organoleptic assessment of the degree of ripening. *Procedure*—Homogenise mechanically 10 g of cheese with 40 ml of 0·5 *M* Na citrate soln. and 80 ml of water. Transfer quant. to a 200-ml flask and dilute to volume. To 100 ml of liquid add 10 ml of 1·41 *N* HCl and dilute to 125 ml. The pH value should be 4·4 ± 0·05. Filter through a Whatman No. 42 paper, dilute an aliquot of the clear filtrate with an equal volume of water and measure the extinction (E) at 274 to 275 mμ (max.) against a blank prepared

from 20 ml of 0·5 *M* Na citrate, 10 ml of 1·41 *N* HCl and 220 ml of water. The coeff. of correlation of E with water-soluble N is 0·958. For the separate determination of tyrosine (*A*) and tryptophan (*B*) as moles per litre of the spectrophotometric soln., measure the extinctions at 270 mμ and 290 mμ; then $A = (0{\cdot}95E_{270} - 1{\cdot}31E_{290})] \times 10^{-3}$ and $B = (0{\cdot}307\ E_{290} - 0{\cdot}020 \times E_{270}) \times 10^{-3}$. Correlation coeff. with water-soluble N are 0·964 and 0·886, respectively. W. H. C. Shaw

4962. **New method of assay of foodstuff dyes. Use of quaternary ammonium salts.** B. Drevon and J. Laur (Lab. d'Intendance des Subsistances, Fac. de Méd. et de Pharm., Lyon, France). *Ann. Falsif.*, 1959, **52**, 155–161.—The dyes are extracted from foodstuffs by combination with a quaternary ammonium salt and extraction with $CHCl_3$ (Auerbach reaction), then separated by chromatography and determined colorimetrically. *Procedure*—The sample is dissolved or suspended in water (proteins are digested with pepsin) and an amount containing 5 to 10 μg of dye is adjusted to pH 9 with Na_2CO_3. Chloroform (10 ml) is added, and, if after stirring for 10 min. it becomes coloured, any basic dyestuffs are determined in it; if it remains colourless it is rejected. The aq. soln. and any ppt. are treated with a large excess of cetyl*cyclo*hexyldimethylammonium bromide, and shaken with $CHCl_3$ for 10 min., and the extraction is repeated until the aq. layer is colourless. The $CHCl_3$ soln. are evaporated at low temp. The residue is dissolved in 0·5 ml of $CHCl_3$ and transferred to bands of Arches 302 paper impregnated with alumina (preparation described), and a chromatogram is run with *n*-butanol - ethanol - water - 20% aq. NH_3 (10:5:5:2). The dyes are identified by their R_F values and estimated by cutting out each spot, eluting with 20% aq. NH_3 and determining the dye photometrically.
E. J. H. Birch

4963. **Analysis of synthetic food colours prescribed in India.** B. R. Roy, A. R. Sundararajan and S. N. Mitra (Central Food Lab., Calcutta). *J. Sci. Ind. Res., India, C*, 1959, **18** (2), 38–40.—A scheme of analysis is given to serve as a routine procedure for the separation, detection, identification and determination of 11 synthetic water-soluble food colours prescribed by the Indian Government. The conventional wool-dyeing test and the colour reactions of the dyed wool and aq. extracts of the colours are used for detection and identification. The identification is confirmed by paper chromatography. The determination is carried out by chromatographing soln. of the dyes in water or *n*-butanol on Whatman No. 1 paper, eluting the chromatogram with 50% aq. ethanol and measuring the eluate in a Beckman spectrophotometer. I. Jones

4964. **Use of a new development chamber for paper-chromatographic detection of subsidiary constituents of colouring matters for foods.** J. Eisenbrand and B. Koether (Chem. Untersuchungsamt f. das Saarland, Saarbrücken, Germany). *Z. LebensmittUntersuch.*, 1959, **109** (3), 218–222.—A description is given of the Koether chamber in which 40 parallel runs can be developed with an ascending solvent on a spirally folded sheet. Most of the substances examined contain subsidiary related colouring matters and substances detectable under u.v. light. During exposure of the chromatograms to daylight for 7 days, some of the previously visible subsidiary spots disappea are sometimes replaced by u.v. fluorescent fluorescent spots may also appear in new pos
P. S. A

4965. **Note on the paper chromatography soluble food colours.** E. Mark and G. G. Mcl (Food and Drug Lab., Dept. of Nat. Healt Welfare, Ottawa, Canada). *J. Ass. Off. Chem.*, 1959, **42** (1), 213–214.—By means procedure of reversed phase chromatog described, quant. separations of a prepared m containing Yellow AB (C.I. Solvent Yellc Yellow OB (C.I. Solvent Yellow 6), Oran (C.I. Acid Orange 20) and Oil red XO (C.I. S Orange 7) were achieved. The loaded pape developed with aq. acetone, and the eth eluates of the separated zones were exa spectrophotometrically. A. A. Eldri

4966. **Artificial food colours. I. Characte properties of permitted water-soluble food c** R. Garcia Olmedo and L. Villanúa (Dept. of Res., Madrid Univ.). *An. Bromatologia*, **11** (1), 9–47.—A classification scheme for based on that of Rota is presented in ext tabular form, and the 25 water-soluble c permitted by Spanish food law are located scheme. Data on chemical and physical prop standards of purity, applications and toxic are presented for each colour.

II. Spectrophotometry of the water-s colours. A. Carballido and L. Villanúa. 1959, **11** (1), 49–108.—Absorption curves a produced for the 25 water-soluble food c permitted in Spain, which include Acid yell (C.I. Acid Yellow 9); tartrazine; quinoline y Sunset yellow FCF (C.I. Food Yellow 3); carmc amaranth; Ponceau 4R (C.I. Food Red 7); throsine BS (C.I. Food Red 14); indigo car auramine; Rhodamine B (C.I. Food Red brilliant croceine; methyl violet; acid violet Food Violet 1); Acrilan violet S4BN (C.I. Food 2) and brilliant green. Graphs of curves gr according to colour, chemical function, cla Rota's classification and chemical class show spectrophotometric identification is possibl yellow, violet or green permitted colours, whi other colours spectrophotometric data can in the interpretation of the results obtained chemical and chromatographic methods.

III. Spectrophotometry of red, orange and water-soluble colours at controlled pH. Carballido and L. Villanúa. *Ibid.*, 1959, **1** 109–178.—Absorption curves for 17 colour cluding Acid yellow G; tartrazine; aura quinoline yellow; Sunset yellow FCF; carmc amaranth; Ponceau 4R; Erythrosine BS; Rhod B and brilliant croceine, at controlled pH ranging from 1 to 11, are presented, and application to the identification of food colo discussed. E. C. Apl

4967. **Determination of sorbic acid. I.** Diemair and K. Franzen (Univ. Inst. f. Le mittelchem., Frankfurt a.M.). *Z. anal. C* 1959, **166** (4), 246–253.—Sorbic acid ma detected by paper chromatography in 1:3: methyl-2-methyleneindoline and the zone ren visible by spraying with methyl *p*-hydroxyben The procedure is unsuited to quant. work be of the poor contrast between the sorbic acid and the general background. For quant. w is recommended that the sample be steam-dis

e extinction of the distillate determined at (pH 5·9) or 262 mμ (pH 4·0). Results are for several proprietary beverages.
W. Diemair and K. Franzen. *Ibid.*, 1959, , 325–333.—Sorbic acid anhydride, formed ling the acid with acetic anhydride, reacts (methylthio)benzothiazolium ethyltoluene-*p*-nate (**I**) to give a green dye. *Procedure*—acid (3 to 5 mg, or the equiv. of sodium or salt) is placed in a flask (500 to 750 ml) ing 100 g of $MgSO_4.7H_2O$, and 100 ml of nd 4 ml of HCl (25%) are added; 90 to of the soln. is distilled. The distillate is if necessary, 2 to 3 drops of HCl are added e soln. is shaken with diethyl ether (5 × The ether is dried with 5 g of P_2O_5 for 0 min., immersing the flask in ice-water, aporated and the residue kept in a desiccator ar. The dry residue is dissolved in 5 ml of anhydride and filtered. An aliquot of the (1 ml) is added to a mixture of dry **I** (30 mg) nhyd. Na acetate (50 mg) and the mixture ersed in an oil bath at 135° for 15 min. The s diluted with methanol to 11 ml, shaken, oled, and the colour is measured after 15 min. mμ, with 0·25-cm cells. T. R. Andrew
B. B. Bauminger

. **Pesticides in food products. V. Deter-on of hydrocyanic acid in food products by graphic polarography.** V. Jedlička and A. (Centr. Res. Inst. of Food Ind., Prague). *. Farm.*, 1959, **8** (3), 138–142.—Hydrocyanic n *M* NH_4Cl as supporting electrolyte yields p incision on the cathodic axis of the curve function $dV/dt = f(V)$ which can be used tection; its depth can be used for the quant. ination of the amount of HCN present 0 μg per ml) after a preliminary distillation. liability of the method was tested by analysing s food products (*e.g.*, milk, vegetables and , blood and urine. J. Zýka

. **Pre-determination of the albumin stability er with 2:6-dichlorophenolindophenol.** K. eisen and G. Wittmann (Inst. f. Gärungs-be, Berlin). *Brauerei, Wiss. Beil.*, 1959, **12** 7–51.—The use of 2:6-dichlorophenolindo-l (**I**) as a precipitant in the rapid pre-ination of the albumin stability of beer has exhaustively studied. Twenty-eight beers tested under standard conditions. To each e was added 0·01 *N* aq. **I** in the proportion l of reagent to 97 ml of beer. The mixtures naintained at 20° for 24 hr. and after a further the turbidities were measured in a nephelo-Results were compared with values obtained rmal air oxidation; beers from the same ry showed good agreement. Values given by nmonium sulphate and Esbach precipitation ds were also compared with those from air ion. Neither of these methods is suitable e pre-determination of albumin stability. In er series of experiments beers were oxidised rious degrees with air, and then tested for in stability with **I**. The results indicate that est gives a satisfactory indication of the ble stability. S. M. Marsh

. **Gravimetric bromimetric determination of value and some quantitative determinations bstitution with a flat weighing glass.** W. rink (Gödecke & Co. A.-G. Chem. Fabrik,). *Fette, Seif., Anstrichmitt.*, 1959, **61** (3), 194–199.—The flat weighing glass consists of a pair of glass plates between which a viscous sample can be pressed into a film. The plates are separated when the sample is being dried or exposed to a gas, and closed for weighing. Apparatus for producing a bromine atmosphere and a procedure for the determination of moisture and of unsaturation in the same sample are described. In sunlight, Br-substitution reactions can be carried out in the same apparatus and their course followed by the increase in wt. G. Burger

4971. **Determination of epoxides in oils and fats.** L. Krull (Margarine Union A.-G., Hamburg-Bahrenfeld). *Fette, Seif., Anstrichmitt.*, 1959, **61** (3), 223–227.—The effect of other oxidation products on the determination of epoxides in fats has been examined and an argentimetric method is proposed which is less susceptible to interference than other methods studied. A soln. of the sample in *iso*propyl alcohol is boiled under reflux with an excess of collidine hydrochloride and the excess of Cl^- is titrated with $AgNO_3$, with Congo red as indicator. The thermal stability of epoxides and the effect of epoxides on the determination of characteristic values of fats were also studied. The epoxide group is stable up to 150°. The carbonyl value and the hydroxyl value are affected, and the peroxide value is not affected, by the presence of epoxides. G. Burger

4972. **Determination of *iso*oleic acids in fats.** J. Pokorný and B. Kakáč (Sch. of Chem. Technol., Prague). *Průmysl Potravin*, 1959, **10** (1), 19–20.—The determination of *iso*oleic acids by the lead salt method yields results that are lower by 1 to 9% than those obtained with the use of the spectrometric method. J. Zýka

4973. **Determination of the yield of oil in winterisation pastes.** G. B. Martinenghi (Studio di Ric. Chim. per Olii, Milan). *Olii Min.*, 1959, **36** (2), 37–40.—Pastes obtained in removing solid fat from olive oils contain ≃70 to 80% of oil. The sample (20 g) is dissolved in hexane (100 ml) cooled to −20°, kept for 4 to 5 min. and then filtered at this temp. The amount of oil in the filtrate is determined by removing the solvent and drying to constant wt. at 110° to 120°. For pastes with different oil contents the conditions are slightly modified. An additional washing with the solvent will give a figure higher by ≃5%, but the value obtained without this treatment is considered to represent the practical oil yield.
L. A. O'Neill

4974. **Separation of lipids by gas - liquid chromatography.** S. R. Lipsky, R. A. Landowne and J. E. Lovelock (Dept. of Internal Med., Yale Univ. Sch. of Med., New Haven, Conn, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 852–856.—By the use of high-efficiency capillary columns in conjunction with the ionisation detection system of Lovelock (*cf. Anal. Abstr.*, 1958, **5**, 3208), the analysis of complex mixtures of the esters of long-chain saturated and unsaturated acids in quantities < 0·1 μg has been achieved. Thus, methyl oleate and methyl elaidate were easily separated on a column (200 ft. × 0·01 in. inside diam.) coated with Apiezon L and operated at 207° with an inlet pressure of 40 p.s.i. of argon.
A. R. Rogers

4975. **Identification of position isomers of some stearic acid derivatives by infra-red spectroscopy. Study of *trans*-6- through -11-octadecenoic acids and corresponding contiguously substituted dihydroxystearic acids in the crystalline state.** Heino

Susi (Agric. Res. Service, U.S. Dept. of Agric., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 910–913.—The absorption spectra of the *trans*-mono-unsaturated fatty acids show a progression of equally spaced strong bands between 1180 cm^{-1} and 1350 cm^{-1}. The number of bands is related to the length of the chain segment next to the carboxyl group and can be used to determine the position of the double bond. The corresponding dihydroxy acids can be distinguished by their spectra in the 800 cm^{-1} to 1200- cm^{-1} region. G. Burger

4976. **Coulometric micro-determination of thiamine hydrochloride.** K. Kalinowski and Z. Sykulska (Dept. of Pharm. Chem., Med. Acad., Lódź). *Acta Polon. Pharm.*, 1959, **16** (2), 111–114.—Thiamine was determined quant. by means of electrolytically generated iodine in an alkaline medium by using the dead-stop method with two platinum electrodes in a specially constructed cell. *Procedure*—To the anode compartment of the cell add 10 ml of the test soln. and 3 ml of 0·1 *N* NaOH. After 5 min. add 10 ml of 10% KI soln. To the cathode compartment add 10 ml of water then equal quantities of the soln. as added to the anode compartment. Cool the cell and its contents to 0° to 2°, switch on the mixing device, then the indicating circuit and finally the generating circuit and record the time from this point. Continue the electrolysis till the presence of free iodine is indicated. Then carry out a blank determination with 10 ml of water instead of the test soln. Calculate the amount of the vitamin in mg from the equation given. One molecule of thiamine reacts with one atom of iodine. The accuracy is ± 0·5% for 3 to 9 mg of thiamine hydrochloride. The method can be applied to the determination of thiamine in tablets and capsules. W. Roubo

4977. **The determination of total riboflavine content in cereals and cereal products.** J. Janicki and E. Kaminski (Inst. f. Landwirtschaftl. Technol., Landwirtschaftl. Hochsch., Poznan, Poland). *Getreide u. Mehl*, 1959, **9** (1), 9–12; (2), 18–21.—Riboflavine was determined in wheat by the fluorescence and lumiflavin methods. By varying the conditions of extraction, riboflavine values from 2·27 to 14·40 μg per g (fluorescence method) and 1·35 to 7·14 μg per g (lumiflavin method) were obtained. The pH of the soln. was found to affect the fluorescence intensity. In both methods, concentration and $CHCl_3$ washing of the extract affected the final results. After the preliminary autolysis, hydrolysis with NaOH instead of H_2SO_4 reduces the recovery. Better results are obtained with a concn. of 2% of original substance than with 5%. In the lumiflavin method, the extraction of the photolysed material with $CHCl_3$ should be continued until the $CHCl_3$ extracts show no fluorescence. It is shown that the photolysis of riboflavine produces lumichrome as well as lumiflavin. The possibility of the formation of lumichrome from riboflavine by the action of micro-organisms present in grain, and of interference in the fluorescence method by xanthopterins, is discussed.
J. V. Russo

4978. **Ion exchangers in pharmaceutical analysis. XII. Separation of pyridoxine and ascorbic acid.** A. Jindra (Dept. Biochem., Charles' Univ., Prague). *Československ. Farm.*, 1959, **8** (1), 15–16.—The separation is carried out with the use of a combination of ion exchangers of different basic strengths. *Procedure*—Place a 3-ml column of a weak basic anion exchanger (Amberlite IR-4b) over a column of strong anion exchanger (Amberlite IRA-401) and mixture of pyridoxine hydrochloride (2 to and ascorbic acid (20 mg) (20 to 30 drops per Wash the columns with H_2O (50 ml), remo upper column, elute the pyridoxine retained lower column with 0·1 *N* HCl (25 ml) and mine photometrically with diethyl-*p*-phe diamine (*cf.* Hrdý and Urbanová, *Anal.* 1958, **5**, 2374). J. Z

4979. **Influence of surface-active compou the polarographic determination of L-ascorbi** J. Davídek and J. Manoušková (Centr. Res of Food Ind., Prague). *Průmysl Potravin*, 1 (2), 96–98.—The presence of 1·25% of gelat 10% of dextran or soluble starch mak measurement of the polarographic wave of a acid slow. To prevent this difficulty and t results caused by surface-active compou method is recommended, in which, for the tion of results by the method of standard ad a soln. of ascorbic acid with added surface compound is used. With this modificati polarographic method yields results which agreement with those by the titrimetric pro J. Z

See also Abstracts—**4722**, Determination **4761**, Determination of F in foods. **4826**, fication of organic acids from alcoholic ferment **4927**, Detection of alkaloids in foods.

Sanitation

Analysis of air, water, sewage, industr wastes, industrial poisons.

4980. **Special indicator tubes for a rapid mination of nitrogen oxides in the air of ind establishments.** L. A. Mokhov, Yu. F. and V. S. Khalturin. *Zhur. Prikl. Khim.*, **32** (2), 452–453.—The construction of indicator tubes, 80 to 100 mm in length an 5 mm in diam., is described. Add a sat ethanolic soln. of H-acid (8-amino-1-naphth disulphonic acid) (20 ml) to silica gel (1 g) for 2 min., evaporate off the ethanol, add a ethanolic soln. of *p*-aminobenzoic acid (20 m again, mix with a 0·1% ethanolic soln. of and grind the indicator mass before drying a suitable consistency. Introduce the ind gel into the indicator tubes to make a 2-mm Above this layer introduce a 2-mm layer o gel followed by a glass- or cotton-wool plu seal the open, broad end of the tube. *Pr for the determination of nitrogen oxides*—Brea the tube at both ends and pass 100 ml of t air through the broad end of the tube so emerges from the cone-shaped end. Compa colour of the indicator with a standard color scale. In the presence of nitrogen oxid indicator changes from pale pink to a cri brown. The indicator is sensitive to 0·0 per litre. K. I

4981. **Infra-red spectral detection of car acids and aldehydes in air-borne particulate** Sawicki and T. R. Hauser (U.S. Dept. of H Educ. and Welfare, Cincinnati, Ohio). *Anal.* 1959, **31** (4, Part I), 523–527.—The reacti various types of carbonyl compounds wit and diethylamine were investigated. In al

rboxylic acid C=O band moves towards a wavelength on reaction with PCl_5 and s a longer wavelength on reaction with amine. Aldehydes lose their C=O band carboxydiarylketonic compounds lose their group on reaction with PCl_5. All other types ɔonyl compounds tested show only minor s in the 5·5 to 6·5-μ region. The application e studies to air-borne particulates has shown arboxylic acids can be identified in the e-soluble fraction and in the acid sub- n. Data for about 40 compounds are listed.
G. P. Cook

. **Micro-determination of dissolved oxygen by rmic syringe.** P. Holland (Power Station, Dorset, England). *Chem. & Ind.*, 1959, (7), 9.—Less than 0·01 p.p.m. of dissolved O in feed water can be determined rapidly and tely by the apparatus and procedure ɔed. To the sample collected (through a in a bottle with a "Suba-Seal" cap are added lowing reagents in succession—(*i*) 40% NaOH (0·05 ml), (*ii*) 40% $MnSO_4$ soln. (0·05 ml), *ii*) conc. HCl satd. with *o*-tolidine (0·1 ml). ıjections are made with an all-glass syringe graduated to 0·1 ml) and stainless-steel . The colour developed is compared in an ɔtiometer, preferably with that given by rly treated samples of de-aerated water ning known amounts of satd. soln. of O. d interfering substances and insol. compounds be removed on a mixed-bed resin column, ı-ionised reducing substances react too slowly ıse interference. Reproducibility is at least d as the sensitivity of *o*-tolidine (0·003 p.p.m.); ent blank is necessary for samples containing xcess of 0·02 p.p.m. W. J. Baker

3. **Portable analyser for determination of ed oxygen in water. Application of rapid- ng mercury electrode.** C. P. Tyler and J. H. mer (Humble Oil & Refining Co., Baytown, *Anal. Chem.*, 1959, **31** (4, Part I), 499–502.—ing a horizontal dropping mercury electrode short drop-time (0·2 to 0·33 sec.) a current is ɔed which is relatively insensitive to agitation sample-flow. The sample flows by gravity gh a cell containing the horizontal cathode mercury pool anode. A voltage of −1·6 V e mercury pool is applied; the circuit includes ɔoammeter (which also serves as a voltmeter) a variable resistance. The total current ɔected for temp. and residual current (found aerating a fresh sample and again recording rrent); 0·1 p.p.m. of O can be determined in ɔH 4·5 to 11); if the salt concn. in the sample ·05 or < 0·001 *M*, 2 ml of reagent (5 g of ı, 2 g of phenol and 0·3 g of NaCl in 100 ml O) is added to each 100 ml of sample. For ıtion, a sample of H_2O containing the reagent ıted for 1 hr. and used 0·5 hr. later; it con- 8·10 p.p.m. of O at 26°.
P. D. Parr-Richard

4. **Polarographic determination of dissolved ı. Study of drop time with rapid-dropping ry electrode.** J. H. Karchmer (Humble Oil & ng Co., Baytown, Tex.). *Anal. Chem.*, 1959, Part I), 502–508.—The rapid-dropping mer- lectrode is suitable for continuously flowing ıs and for amperometric titrations with con- ıs stirring. The effect of drop-time (*t*) and g on the diffusion current of O in water is sed; a single capillary electrode mounted vertically, then horizontally, and finally scratched at the orifice and mounted vertically, was used for the tests. For the same flow-rate of mercury, *t* was 0·8 to 4 sec., 0·2 to 1·0 sec., and 0·025 to 0·07 sec., respectively. For very small drop-times, the current does not obey the Ilkovič equation; stirring affects the current less as *t* decreases and the rate of mercury-flow increases. When *t* is < 0·2 sec. and the salt concn. is high, an anomalous current is produced. Simple equations relating *t* and mercury flow-rate can be used for the selection of suitable capillaries.
P. D. Parr-Richard

4985. **Polarographic determination of oxygen. Anomalous current encountered with rapid-dropping mercury electrode.** J. H. Karchmer (Humble Oil & Refining Co., Baytown, Tex.). *Anal. Chem.*, 1959, **31** (4, Part I), 509–513.—In the determination of O in water an anomalous high current is obtained when the salt concn. is > 0·05 *N*; the effect is accentuated as the drop-time of the mercury decreases and the total current increases, and is related to the "water current." For the horizontal capillary used, the addition of 0·075% by wt. of gelatin suppressed the effect completely, giving curves similar to those found with drop-times of 3 to 6 sec., or in the absence of high salt concn.
P. D. Parr-Richard

4986. **B.D.H. indigo carmine method for determining dissolved oxygen in boiler feed water.** British Drug Houses, Ltd. *Chem. Age*, 1959, **81**, 618.—A simple colour-comparison method for the determination of ≯ 0·12 ml of O per litre is described. The reagents required are indigo carmine - glucose tablets, KOH soln. (51·5 g in H_2O to make 100 ml) and glycerol. A modified Nessler cylinder (containing a reagent tube sealed with a glass ball) is filled with the sample, stoppered and inverted, so that the reagent flows into the water. The colour is compared with a standard disc in a Lovibond Nessleriser. Nickel, Cu^{II} or Zn (1 p.p.m.), Fe^{III} (3 p.p.m.) and small amounts of SO_3^{2-} or hydrazine do not interfere. Hydrazine can be caused to react with *p*-dimethylaminobenzaldehyde to give a red or yellow colour in conc. or dil. soln., respectively, which can be determined spectrophotometrically (*cf.* Watt and Chrisp, *Brit. Abstr. C*, 1953, 161); urea and semicarbazide do not interfere.
P. D. Parr-Richard

4987. **Determination of calcium, magnesium and total hardness by automatic spectrophotometric titration.** H. V. Malmstadt and T. P. Hadjiioannou (Univ. Illinois, Urbana, U.S.A.). *J. Amer. Wat. Wks Ass.*, 1959, **51** (3), 411–417.—Calcium is determined in the presence of Mg at pH 13, with Calcon (C.I. Mordant Black 17) as indicator. The total amount of Ca and Mg is determined at pH 10 with Eriochrome black T (C.I. Mordant Black 11) as indicator. Both titrations are automatically terminated at their end-points. Interference by heavy metals is prevented by adding a few drops of KCN soln. The results from more than 1000 samples with total hardness from 0·5 to 600 p.p.m. were reproducible and accurate. The automatic titration takes from a few seconds to a few minutes, according to the hardness.
O. M. Whitton

4988. **Ion exchange - spectrographic determination of arsenic and phosphorus in river water.** R. Ko (General Electric Co., Hanford River Products Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-59008, 1959, 14 pp.—Concn. as low as 0·001 p.p.m. were determined with a

precision of ± 0·001 p.p.m. (95% confidence level for a single measurement). One litre of sample was concentrated by anion exchange. Arsenic and P were eluted with *M* HNO_3 and determined in the eluate by d.c. arc excitation, with V as the internal standard. NUCL. SCI. ABSTR.

4989. **Separation and determination of microgram quantities of molybdenum in natural waters.** Ken Sugawara, Motoharu Tanaka and Shiro Okabe (Chem. Inst., Fac. of Sci., Nagoya Univ., Chikusa-ku, Nagoya). *Bull. Chem. Soc. Japan*, 1959, **32** (3), 221–222 (in English).—The Mo is first co-pptd. with either hydrated manganese oxide or ferric hydroxide, and then extracted with *n*-butanol - $CHCl_3$ as the thiocyanate and subjected to spectrophotometry at 475 mμ. The pH of the soln. is adjusted to 3·8 to ensure quant. co-pptn. of Mo and to give nearly 100% recovery. Co-pptn. with manganese oxide is preferred to co-pptn. with ferric hydroxide since the ppt. is quickly and easily filtered off and is more readily soluble in dil. HCl containing a small amount of H_2O_2. The optimum pH range for quant. pptn. is also wider. I. JONES

4990. **Analysis of strongly mineralised waters.** R. Marcille. *Chim. Anal.*, 1959, **41** (3), 105–109.—Waters from dry regions may change in composition during transit owing to bacterial action; thus, SO_4^{2-} form S^{2-}, and NO_3^- form NH_4^+. Sample bottles and corks must be clean and the addition of $CHCl_3$ (5 ml per litre) is recommended as a preservative. The determination of dry residue, alkalinity, NO_3^- and K^+ is discussed. On heating the dry residue to 250° to dehydrate $CaSO_4.2H_2O$, some Cl^- are lost if $MgCl_2$ is present, though $Ca(HCO_3)_2$ helps to prevent this. Such residues re-hydrate very rapidly. Alkalinity must be determined on the natural water, not on the dry residue, and contamination by gas heating must be avoided when SO_4^{2-} are to be determined. For the determination of K^+ as $KClO_4$, excess of $HClO_4$ must be present during the evaporation, and the ethanol (satd. with ClO_4^-) must be acid (2 ml of $HClO_4$ per litre). A second evaporation is recommended to convert all NaCl into $NaClO_4$. The classical method of grouping elements in the dry residue by combining results for CO_2, SO_3^{2-}, Cl^- and NO_3^- with those for Ca^{2+}, Mg^{2+}, K^+ and Na^+ is recommended, and examples are given. P. D. PARR-RICHARD

4991. **Application of an automatic titrator to sewage analysis.** J. C. Crowther (Houldsworth Sch. of Appl. Sci., Univ., Leeds, England). *Chem. & Ind.*, 1959, (10), 327–328.—Automatic potentiometric titrations of NH_3 and "oxidised N" can be completed in < 1 min. by connecting two titration units with one titrator by means of a change-over switch; the end-point is taken at pH 4·70. The burette tip is placed so that stirring carries the titrant towards the indicator electrode; the automatic change-over from fast to slow delivery can be adjusted to occur a few drops before the end-point. The apparatus consists of a Pye Universal pH meter, a Pye automatic titrator, and a switching unit. P. D. PARR-RICHARD

See also Abstracts—**4758**, Determination of F in air. **4761**, Determination of F in water. **4875**, Determination of Tl in air. **5008**, Determination of demeton in air. **5073**, Alarm for detection of toxic gases.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

4992. **Rapid determination of readily s magnesium in soils.** B. E. Beater and R. R. (Sugar Expt. Station, Mount Edgecombe, S. Africa). *Chemist Analyst*, 1959, **48** (1), 10 The colour produced by an acid extract with Brilliant yellow (C.I. Direct Yellow measured absorptiometrically and compared standards. *Reagent*—Dissolve 0·01 g of Br yellow in 50 ml of methanol. Mix 30 g of poly(vinyl alcohol) soln. with 50 ml of wate add it with stirring to 900 ml of satd. lime Add the Brilliant yellow soln., stir and *Procedure*—Shake 2·5 g of soil (air-dry, mesh) with 25 ml of 0·05 *N* H_2SO_4 for 1 min. through an asbestos pad between two filter- with suction. To 1 ml of filtrate add 25 reagent with rotation, set aside for 20 mir measure the extinction with a green filter (56 Compare the result with those for standards ob by adding 1 drop of 0·08% aq. $Al_2(SO_4)_3$. soln. to 1 ml of suitably diluted Mg soln. (3 of $MgSO_4.7H_2O$ made up to 1 litre with (H_2SO_4) and treating as described above. Wh soil contains enough extractable Al to cause c ness, treat 5 ml of soil extract with 6 drops o aq. $(NH_4)_2SO_4$ soln. and 5 ml of satd. lime set aside for 10 min., filter, and treat 2 ml filtrate as described above. The result sufficiently close to those by titrimetric and photometric methods to render the method su for agricultural advisory purposes. The extract of the soil can be used for flame-photo determinations of Ca and K. R. E. Ess

4993. **Rapid method for the determinati total sulphur in soils and plants.** B. Butter E. M. Chenery (Dept. of Agric., Kawanda Sta., P.O. Box 265, Kampala, Uganda). *A* 1959, **84**, 239–245.—The method is turbidin Standard soln. are prepared from an aq. so $MgSO_4.7H_2O$ (50 μg of S per ml) acidified acetic acid, 25% HNO_3 and H_3PO_4. B chloride crystals are added to each soln. and a specified number of inversions at specified intervals, clear acacia gum soln. is added liquid is adjusted to a known vol., inverted according to a specified procedure and its e tion is measured in a Spekker absorptiome 490 mμ. The calibration graph is linear. soil sample is evaporated in an electric ov 70° with 2 ml of $Mg(NO_3)_2$ soln., prepared Specpure magnesium dissolved in HNO_3 (2 500 ml), the residue is heated overnight in a sta steel oven at 300° and then digested with HN a water bath. The cooled digest is adjusted known vol. and filtered. An aliquot is t with 50% acetic acid and H_3PO_4 and the tur is developed as with the standard soln. A sl modified procedure is used with plant ma The same batch of $BaCl_2$ crystals should be for samples and standards, a reagent bla necessary and determinations should be ma duplicate as occasional determinations may error by > 10%. A. O. Jo

4994. **Spectrophotometric detection of d diamide in calcium cyanamide and in soil** Pawlik (Stuttgart-Hohenheim, Inst. f. Pfla schutz d. Landwirtschaftl. Hochsch. Hohen *Naturwissenschaften*, 1959, **46** (6), 203.—Th

on spectrum of dicyandiamide shows a m at 215 mμ which can be used for the n of dicyandiamide in aq. extracts from l calcium cyanamide in the presence of ne, dicyandiamidine and urea, and for its nation within 12% at a concn. of 5 mg in soil. Nitrates interfere. G. BURGER

Composition and manurial value of poultry straw-droppings composts and deep litter. oduction: experimental materials, methods oling and analysis. J. Tinsley and T. Z. wski (Dept. of Soil Sci., Univ. of Aberdeen). *Food Agric.*, 1959, **10** (3), 145–150.—Evalua- various manures has been made by deter- their N, P and K contents; precautions ken against loss of N as NH_3. A fresh sample (20 to 30 g) is blended with H_2O suspension inverted rapidly over a metal livider to give four 100-ml fractions, which n weighed. Total N is determined on one , after reduction with 1 g of Devarda's alloy ml of 40% NaOH soln. (an acid-soaked eventing loss of NH_3), by Kjeldahl digestion $_2SO_4$; NH_3 is distilled and titrated in the ray. To another fraction is added 0·1 *N* (25 ml) and H_2O to make 250 ml; after aside overnight, soluble N is determined on ate. Total soluble N is found by reduction estion as before, soluble N excluding NO_3^- by $FeSO_4$ and conc. H_2SO_4 to destroy NO_3^- ligestion, and NH_3 by distillation at pH 9. organic N and NO_3^- are found by difference. cid is determined by extraction with and titration in LiCl - HCl soln. with $_2.2(NH_4)_2SO_4$ soln. (*Analyst*, 1957, **82**, 110). isture of fresh samples is found by heating for 24 hr., and the pH after soaking a fresh for 1 hr. in H_2O. The ash content is ed by charring 5 g of dry material and overnight at 500°; the C (%) is taken 8% of the organic matter lost on ignition. et-ashing 1 g of dry material with HNO_3 - P in the diluted digest is determined by olybdovanadophosphate method (Hanson, *Food Agric.*, 1950, **1**, 172). For K, 1 g of nple is moistened with H_2SO_4, ashed at 520° and extracted with dil. HCl; K is ed by flame photometry. Experimental re discussed and the precision is shown to be P. D. PARR-RICHARD

Improved microbiological method for determination [in forages]. R. M. Hill, H. , W. E. Engelhard and M. Bogard (Dept. of n. and Nutrition and Dept. of Bact., Univ. raska, Lincoln, U.S.A.). *J. Agric. Food* 1959, **7** (4), 261–264.—The combined tic and colorimetric method developed by and Evans for the determination of NO_3^- t materials (*J. Biol. Chem.*, 1953, **202**, 655) dified by Garner *et al.* (*Anal. Chem.*, 1956, 9) has been further improved by the use of o-organism (*Pseudomonas oleovorans* ATCC 32) which does not produce nitrite reductase es not have a nutritional requirement for The maintenance of the stock culture and aration for the assay are therefore simplified, incubation time in the assay is less critical. od precision is attainable, and in most cases lts compare favourably with those obtained nodified Devarda method. S. C. JOLLY

4997. **Simplified determination of molybdenum in plant material by 4-methyl-1:2-dimercaptobenzene (dithiol).** J. B. Bingley (Div. of Animal Health and Production, C.S.I.R.O., Parkville, Victoria, Australia). *J. Agric. Food Chem.*, 1959, **7** (4), 269–270.—A rapid, sensitive and accurate colorimetric method is described, capable of determining 0·1 to 5·0 μg of Mo in plant material without preliminary separation. The presence of sufficient Fe is essential for max. colour development, but Cu in the amounts usually present in plant material (> 30 μg per g) does not interfere. *Procedure*—Wet-oxidise the sample (1 g) with H_2SO_4, HNO_3 and $HClO_4$, evaporate the soln. twice to fuming, cool, dilute with 4 to 5 ml of water and boil vigorously for 15 sec. Add 0·25 ml of Fe soln. [9·1 g of $(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$ in 100 ml of 2% H_2SO_4], dilute to 25 ml with water, add 0·25 ml of KI soln. (50% w/v), swirl occasionally during 10 min., discharge the iodine colour with $Na_2S_2O_3$ soln. (10% w/v), and add 0·25 ml of tartaric acid soln. (50% w/v) and 2 ml of dithiol reagent (dissolve 1 g in 500 ml of 1% NaOH soln. warmed to 38°, and add thioglycollic acid until a faint turbidity persists; store in a refrigerator and use within 1 month). Shake vigorously for 30 sec. and, after 30 min., add 5·0 ml of *iso*amyl acetate (boiling-range 136° to 142°) and again shake vigorously for 30 sec. Centrifuge the organic layer to remove water droplets, measure the extinction of the soln. at 680 mμ, and determine the amount of Mo by reference to a calibration curve prepared by treating similarly a standard soln. of Mo (prepared from molybdenum metal to contain 1 μg per ml).
S. C. JOLLY

4998. **Colorimetric determination of 3:5-dinitrobenzamide in feeds by the Janovsky reaction.** H. F. Beckman and J. De Mottier (State Chem. Lab., Texas Agric. Exp. Sta., College Station, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (4), 280–282.—The Janovsky reaction for dinitro compounds has been adapted to the rapid routine determination of microgram amounts of 3:5-dinitrobenzamide (**I**) in poultry feeding-stuffs. The source of the feeding-stuff had little effect on recoveries of **I** (97·4 to 103·5% for 12 different materials), and 4-hydroxy-3-nitrophenylarsonic acid, NN′-di-(*m*-nitrobenzenesulphonyl)ethylenediamine, N^4-acetyl-N^1-(*p*-nitrophenyl)sulphanilamide and nitrophenide [di-(3-nitrophenyl) disulphide] did not interfere. *Procedure*—To 2·0 g of the feeding-stuff (containing 0·025% of **I**) in a 100-ml flask add anhyd. methanol to the mark, shake for 30 min. and filter. Evaporate a 20-ml aliquot to dryness, dissolve the residue in 10 ml of warm acetone, and rinse the vessel with 5 ml of conc. aq. NH_3, followed by 10 ml of acetone. Dilute the combined acetone soln. and rinsings to 25 ml with acetone, mix well, and after 45 min. in the dark measure the extinction at 580 mμ. For feeding-stuffs containing higher percentages of **I**, suitably dilute the methanolic extract so that a 20-ml aliquot contains 120 μg of **I**. Prepare a standard curve by adding 5 ml of conc. aq NH_3 to 20-ml aliquots of acetone soln. containing 0 to 10 μg of **I** per ml, and measuring the extinction as for the test soln. S. C. JOLLY

4999. **Determination of arsenic in some insecticides.** D. Pirtea and E. Antonescu (Chem. Res. Centre, Acad. R.P.R., Bucharest). *Acad. R.P.R., Stud. Cercet. Chem.*, 1959, **6** (3), 445–453.—The method is based on the reduction of the arsenic

compound to AsH_3, followed by oxidation to arsenate with H_2O_2 in the presence of KOH and titration of the As iodimetrically. H. Sher

5000. **Determination of HCH [hexachlorocyclohexane] and DDT in commercial preparations.** F. Strache (Hyg. Inst., Hamburg). *Fette, Seif., Anstrichmitt.*, 1959, **61** (3), 186–187.—On saponification with ethanolamine, HCH yields three Cl^- per mol. and DDT yields one Cl^-. With metallic Na in *iso*propyl alcohol all five Cl^- are split from DDT, and, similarly, all six Cl^- are split off from HCH. Thus two Cl^- determinations (by addition of an excess of standard $AgNO_3$ soln. and back-titration of the filtrate) measure the DDT and HCH separately. The reaction with ethanolamine is carried out in 2-ethoxyethanol soln. on a water bath. The reaction with Na is carried out by boiling the soln. in *iso*propyl alcohol with Na for 30 to 40 min. under reflux. G. Burger

5001. **Gas chromatography of pesticides.** D. M. Coulson, L. A. Cavanagh and J. Stuart (Dept. of Chem., Stanford Res. Inst., Menlo Park, Calif., U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (4), 250–251.—Lindane, parathion, aldrin and *pp'*-DDT were recovered quantitatively, with no evidence of decomposition, after passage through a gas-chromatographic column at 220° to 280° with He as the carrier gas, followed by absorption in CS_2 and subjection to i.r. analysis. Dieldrin, malathion and demeton can also be separated from pesticide mixtures. Toxaphene and chlordane gave complex chromatograms indicative of a mixture of components. The technique should be useful for the 'clean-up' and separation of pesticides in vegetable extracts. S. C. Jolly

5002. **A fly bio-assay for the determination of organic phosphate pesticides.** D. F. McCaulley and J. W. Cook (Div. Food, Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (1), 200–207.—The method is described, and tabulated data show the effect of parathion, parathion-methyl, EPN (O-ethyl O-*p*-nitrophenyl phenylphosphonothioate), malathion, Systox, diazinon, Guthion [OO-dimethyl S-(4-oxo-3H-1:2:3-benzotriazine-3-methyl) phosphorodithioate], Trithion [S-(*p*-chlorophenylthio)methyl OO-diethyl phosphorodithioate] and Phosdrin (1-methoxycarbonyl-1-propen-2-yl dimethyl phosphate, 60% technical) individually on house-fly mortality and on the *in vivo* inhibition of fly cholinesterase. A close relationship is indicated. Lindane, DDT, isodrin and toxaphene showed no effect on cholinesterase. A. A. Eldridge

5003. **Comparison of four cholinesterase methods of analysis for organic phosphate pesticides.** G. Yip and J. W. Cook (Div. Food, Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (1), 194–197.—The inhibition of cholinesterase was measured by the electrometric, titrimetric, manometric and colorimetric methods, with Systox as inhibitor. For 50% inhibition there was a maximum difference of $\pm$ 15% between the results, which are illustrated graphically. It was observed that the technical Systox employed became more potent with lapse of time. A. A. Eldridge

5004. **Direct colorimetric analysis of esterase-inhibiting insecticides with indo acetate.** T. E. Archer and G. Zweig (P Residue Res. Lab., Univ. of California, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (3 181.—Residues of the anticholinesterase cides, Sevin (1-naphthyl methylcarbamate), [OO-diethyl S-(ethylthio)methyl phosphoro ate], Guthion [OO-dimethyl S-(4-oxo-3H- benzotriazine-3-methyl) phosphorodithioate Trithion [S-(*p*-chlorophenylthio)methyl OO- phosphorodithioate] are determined colorime by measuring the product of the hydrol indophenyl acetate at pH 8·0 by the su enzyme. The effects of source of enzyme, concn., temp. and pre-incubation time are re Crystalline bovine erythrocyte acetylcholine was a satisfactory source of enzyme; enzyme human and horse sera were relatively insens Sevin, to which bee-brain cholinesterase, other hand, was particularly sensitive. Res this colorimetric method agreed well with obtained potentiometrically and by paper c tography. M. D. Ande

5005. **Cholinesterase-inhibition method of a for organic phosphate pesticides: effect of e inhibitor reaction time upon inhibition.** Nesheim and J. W. Cook (Div. Food, Fo Drug Admin., Dept. of Health, Educatio Welfare, Washington, D.C., U.S.A.). *J. A Agric. Chem.*, 1959, **42** (1), 187–193.—The phorylation or inhibition of the enzyme was f by time studies in order to ascertain the op degree of enzyme phosphorylation within a able and practicable incubation time. The formation of the enzyme - phosphoryl comp stability, and the reactivation, if any, enzyme, are involved. Studies on the ef time of incubation of Phosdrin (1-me carbonylprop-1-en-2-yl dimethyl phosphate technical) or Systox [mixture of OO- S (and O)-2-(ethylthio)ethyl phosphorothioa of oxidised diazinon, malathion, EPN (O-eth nitrophenyl phenylphosphonothioate), par methyl and parathion with human plasma esterase show that the cholinesterase is s to different quantities of the various pesticid that the rates of reaction vary consid Incubation for 30 min. is recommended. A. A. Eldr

5006. **Detection of phosphorothioic acid** R. Fischer and N. Otterbeck (Inst. f. Phar d. Univ. Graz). *Sci. Pharm.*, 1959, **27** (1), Develop an extract of the sample, or altern a sulphur-containing saponification produc either acetone - H_2O - 12% aq. NH_3 (130:6 methanol - H_2O - 1% aq. NH_3 (19:1:1) method of "test-tube chromatography" (and Otterbeck, *Ibid.*, 1957, **25**, 242). Mom dip the chromatogram in a soln. containing azide and iodine. Sulphur-containing com are revealed as colourless spots on a brow ground. The sensitivity is 1 or 2 μg. V R_F are tabulated for parathion, diazinon, Chl [O-(3-chloro-4-nitrophenyl) OO-dimethyl p phorothioate], malathion, metaSystox (de methyl) and thiometon (OO-dimethyl thioethyl phosphorothiolothionate) and their fication products. A. R. Ro

5007. **Extraction and identification of pa and diazinon from lettuce.** R. E. Mode J. W. Cook (Div. Food, Food and Drug

ngton, D.C., U.S.A.). *J. Ass. Off. Agric.*, 1959, **42** (1), 208–211.—The use of methyl le for extraction of the pesticides is recom-ed. The extract is diluted with water and esticide transferred to pentane. Interfering extractives are removed on a column of followed by one of MgO - Celite. The pesti-s finally separated by paper chromatography, 4% of mineral oil in diethyl ether as the nary phase and aq. ethanol as the mobile , the paper being then exposed to an atmo-e of Br in CCl_4 and sprayed with fluorescein Recoveries of parathion and diazinon of 92 7% are reported. A. A. ELDRIDGE

8. **Identification of Systox [demeton]. Use ampling-tube techniques in toxicological istry.** K. Grosskopf. *ChemikerZtg*, 1959, **83** 15–117.—The use of small sampling tubes ining adsorbent for the detection and deter-tion of toxic gases is outlined and their ntages are considered. A procedure of this nas been developed for the detection and semi-t. determination of demeton, which is based e addition reaction of the thioether sulphur $AuCl_3$, the product being developed with an oln. of chloramine T to give a highly coloured lex. The sampling tube (illustrated) contains rier impregnated with $AuCl_3$, and above this ampoule containing the developing reagent. easured quantity of air is drawn through the by means of a gas-detection pump and the ule is then broken at a marked point so that eveloping soln. wets the carrier. The appear-of a red - orange ring on the adsorbent ates the presence of demeton. The method is tive to 1 μg, and by taking a sample of 2 litres r it is possible to detect a concn. of 0·5 mg cu. metre. The amount of demeton can be ated approx. by comparing the length of the red ring with a series of standard tubes pre-l from soln. containing known amounts of ton. The method may also be applied to the mination of demeton in organic materials by ing a pentane extract of the material (*e.g.*, s sprayed with insecticide) through the tube. cal results are tabulated. S. M. MARSH

09. **Photofluorimetric method for determina-of Co-Ral [O-(3-chloro-4-methylumbelliferone) iethyl phosphorothioate] [Bayer 21/199] residues imal tissues.** C. A. Anderson, J. M. Adams D. MacDougall (Chemagro Corp., Kansas City, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (4), 259.—A highly sensitive method is described he determination of traces ($\simeq$ 0·02 p.p.m.) of pesticide and/or its oxygen analogue in a ty of animal tissues. Extracts of the tissues) are purified by solvent extraction and matography, and the pesticide is then hydro-to a more highly fluorescent decomposition uct (3-chloro-4-methyl-7-hydroxycoumarin). fluorescence of the soln. is measured at 410 mμ activation at a wavelength of 330 mμ. With 1 p.p.m. of pesticide, the average deviation the mean was $\simeq$ 10%; with higher concn. precision was slightly better. S. C. JOLLY

10. **Paper-chromatographic identification of [1-(1-naphthyl)-2-thiourea].** K. Fischer. *Arch. inol.*, 1957, **120** (3–4), 72–73.—Antu can solated in the acid ether extract of the ical Stas-Otto procedure. The ether is evapor-to dryness and the residue is dissolved in methanol. The soln. is spotted on S. & S. No. 2043 filter-paper. This paper is then prepared for overnight ascending chromatography with ethanol - amyl alcohol - aq. NH_3 (2:2:1). After air-drying the paper it is sprayed with a saturated soln. of Br in 10% NaOH. Pure antu gives a violet spot at R_F 0·79. As little as 30 μg can be identified.

CHEM. ABSTR.

See also Abstracts—**4908**, Determination of organic acids in grass. **4960**, Rotenoids in milk.

5.—GENERAL TECHNIQUE AND APPARATUS

General

5011. **New type of semi-automatic pipette.** I. Heyduk (VŽKG, Ostrava, Czechoslovakia). *Chem. Průmysl*, 1959, **9** (4), 196–197.—The sample is sucked by means of a water-pump into the pipette, the manipulation of the three-way tap of which permits the filling of the pipette and the measurement of the vol. The arrangement is suitable for measuring toxic soln. J. ZÝKA

5012. **Improvements in or relating to pipettes.** M. C. Sanz. Brit. Pat. 815,199, date appl. 21.6.57. Date appl. Switzerland 10.9.56; date appl. U.S.A. 11.4.57.—A capillary tube having a passage capable of retaining a predetermined vol. of liquid by capillary action extends into a chamber which has its lower portion in communication with a vessel containing a reserve of the liquid. Means are provided (*e.g.*, by compressing the vessel, which is made of deformable plastic) for causing the liquid in the reserve vessel to rise into the chamber and submerge the adjacent end of the tube whereby the capillary passage is filled with liquid. The capillary tube may be of polyethylene to facilitate calibration by cutting off the end until the predetermined vol. is contained between the menis-cusses. J. M. JACOBS

5013. **A simple reservoir burette for alkaline solutions.** H. F. Holden (Walter and Eliza Hall Inst. Med. Res., Melbourne, Australia). *Aust. J. Exp. Biol. Med. Sci.*, 1959, **37** (1), 107–108.—A simple and cheap reservoir burette that protects $Ba(OH)_2$ soln. from atmospheric CO_2 is described and illustrated. N. E.

5014. **A recording titrator.** R. C. Williams, R. S. Ruffin and L. A. Mounter (Dept. of Biophys., Med. Coll. of Virginia, Richmond, U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 611–615.—The instrument described has been designed for the titration of acidic and basic groups liberated in hydrolytic enzyme reactions. It is simple, rugged, compact and accurate, and needs no external accessories apart from a water bath for temp. control. The instrument can be used with small samples and is completely free from drift. K. A. PROCTOR

5015. **Apparatus for titrations using ultra-violet light.** D. H. Wilkins (Res. Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Talanta*, 1959, **2** (1), 88–89.—The apparatus is a wooden box painted dull black inside and containing three 8-W fluorescent lamps (General Electric F8T5B1B). A burette passes through the top of the box and a

viewing slot is provided in the side. The contents of the titration vessel are stirred by a magnetic stirrer inside the box. W. T. CARTER

5016. **Acoustic gas analyser.** L. Molyneux (Dept. of Anaesthetics, Univ. of Durham, England). *J. Sci. Instrum.*, 1959, **36** (3), 118–120.—The concn. of halothane ($CF_3CHBrCl$) or $CHCl_3$ vapour (0 to 4%) in a mixture with air and oxygen is measured with an accuracy of $\pm$ 0·25% by comparing the velocity of sound in the mixture before and after removal of the vapour with activated charcoal. An all-transistor electronic circuit is described. G. SKIRROW

5017. **Improved method and apparatus for determining and controlling the moisture content of dielectric materials [particularly textile fibres].** Commonwealth Sci. and Indust. Res. Organisation. Brit. Pat. 815,000, date appl. 30.8.56; date appl. Australia 31.8.55.—The material is located between the plates of a test condenser into which voltages of different frequencies are fed simultaneously. The resulting voltage changes, which are dependent on the moisture content of the material, are converted into logarithmic voltages. These logarithmic voltages are subtracted to obtain a voltage which is a function of the ratio of the change in voltage drops across the plates of the test condenser, corresponding to each feed frequency when the material is placed between the plates. J. M. JACOBS

5018. **Apparatus for the accurate determination of oxygen in a small sample of gas.** J. Muzyczuk (Dept. Phys.-Chem. Anal., Inst. Mining Res., Mikołów, Poland). *Chem. Anal., Warsaw*, 1959, **4** (1–2), 365–372.—A glass apparatus is described and illustrated for the determination of the O content of a 10-ml sample of air or other gas, in the range up to >20% (v/v), with an accuracy of $\pm$0·02%. The reduction in pressure caused by the reaction of phosphorus with O is allowed to suck water from a micro-burette, and the percentage of O is calculated from the vol. of water displaced. The manipulation and maintenance are simple and the reaction time is $\geqslant$ 8 min. Data are provided for the calibration of the burette and the calculation of corrections, which are usually small. W. B. MIASKOWSKI

5019. **Simple expedient for evaporating small amounts of solvent.** R. L. Clements (Dept. of Plant Biochem., Univ. of California, Riverside, U.S.A.). *Chemist Analyst*, 1959, **48** (1), 20.—The solvent (3 to 10 ml) is contained in a vial closed with a neoprene stopper carrying a glass tube. The tube is connected to the vacuum line by a length of Tygon tubing (0·25 in. outside diam.), by means of which the vial is suspended from a clamp in a 250-ml beaker, preferably of plastic or lined with some resilient material, which is placed on a magnetic stirrer, and is filled with water at a suitable temperature. When the stirrer is started and the vacuum applied, the vial is swirled at a frequency depending on the length of tubing between the clamp and the vial, the best position being found by experiment. By this means, 5 ml of light petroleum in a 7·5-ml vial can be evaporated in 2 min. at 30°. The apparatus is illustrated. R. E. ESSERY

5020. **Useful fraction collector for small-scale vacuum distillations.** D. W. S. Evans (Univ., Nottingham, England). *Chem. & Ind.*, 1959, (7), 219–220.—The glass apparatus described and illustrated is a modification of the Kon rectangle will give a clean separation of 2 to 3 ml of disti Advantages claimed are (*i*) all traces of a fra can be removed from the run-off tube before necting a second receiver, (*ii*) this receiver then be evacuated without affecting the distilla and (*iii*) only two simple double-oblique stopcocks are required. W. J. BAK

5021. **Simple micro-Kjeldahl apparatus.** Ta Asami (406th Med. Gen. Lab., Camp Zama, Ja *Anal. Chem.*, 1959, **31** (4, Part I), 630.—With steam-distillation apparatus described and strated, alkali was never distilled over on st heating unless foam reached the outlet to condenser. If foam was produced it tended t destroyed before reaching the outlet. G. P. Co

5022. **Vacuum sublimation device for the se tion and purification of small quantities of stances.** G. Schmidt (Inst. f. gerichtl. Med., U Erlangen, Germany). *Mikrochim. Acta*, 1959, 406–418.—The apparatus described and illustr is based on the principle of temperature grad (*cf.* Askew *et al.*, *Proc. Roy. Soc., B*, 1930, 748; Behrens and Fischer, *Naturwissenscha* 1954, **41**, 13). It is possible to carry out in a si operation the continuous fractionation of se substances which have different sublimation haviours. With the size of apparatus ch ($\simeq$ 45 cm long $\times$ 5 cm diam.) amounts of stance from a few μg to $\simeq$ 30 mg may be han As the various concentric tubes are transpare is possible to follow the course of the sublima By the use of a removable plastic film for the de tion of the sublimate, the fractions may be recov without alteration of their crystalline state. D. F. PHILLI

5023. **Expedient for micro-filtration.** J. Corliss (U.S. Army Chem. Warfare Lab., A Chem. Center, Md.). *Chemist Analyst*, 1959 (1), 16.—A 2-ml hypodermic syringe is inse by means of a 22-gauge hypodermic needle, the rubber stopper connecting the siphon tube filter-tube of the apparatus of Niederl and Nie A variable air-space is thus introduced into system and, by manipulation of the plu particles can be dislodged from the walls of pressure tube, attainment of solubility equil is hastened so that volumes of wash-liquor ca controlled, and clogged filter-tubes can be fr A diagram is given. R. E. ESSE

Chromatography, ion exchange, electrophoresis

5024. **Apparatus for chromatography permi concentration of the constituents.** R. Audran G. Reutenauer (Lab. Kodak-Pathés, Vince France). *Bull. Soc. Chim. France*, 1959, **26** 46–51.—An apparatus is described consisting base plate cooled with water and carrying a circ trough, and a central polished portion acting reflector to a small heating spiral. A cone n from a 145° sector of a circular paper (9·5 cm di is placed with its edge in the trough and p upwards, and the assembly is covered with a g cone which can be closed by a tap or conne to a vacuum pump. As the solutes ascend the p with the solvent they are separated partly

atography and partly by crystallisation owing poration of the solvent and are concentrated s evaporation and by the narrowing arc of The evaporation is promoted by the heater. adaptation replacing the heating spiral by sulated lead, separation by electrophoresis e effected. Examples of organic and in- c separations are given. E. J. H. Birch

5. Variable gradient device for chromato-y. E. A. Peterson and H. A. Sober (Lab. of em., U.S. Dept. of Health, Education and re, Bethesda, Md.). *Anal. Chem.*, 1959, **31** 57–862.—In this device, for which construc- details are given, small changes in specific ns of the elution gradient are possible with- ay physical change in the apparatus. Exact ation of data can be obtained.
K. A. Proctor

3. Improved method of heating chromato-c columns. L. R. Bunney, R. Phillips and Freiling (U.S. Naval Radiolog. Defense Lab., rancisco, Calif.). *Anal. Chem.*, 1959, **31** (5), -In the method described, a heating jacket ctrically conducting Pyrex-glass tubing is The electrically conductive film is on the e of the tubing and is chemically inert, very nd transparent. The max. operating temp. °.
K. A. Proctor

7. Centrifugal force in paper chromatography ectrophoresis. H. J. McDonald, L. P. Ribeiro . J. Banaszak (Loyola Univ., Chicago, Ill., .). *Anal. Chem.*, 1959, **31** (5), 825–829.— ple and effective apparatus is described for plication of centrifugal force to paper chroma- hy and ionography on paper discs rotating orizontal plane. The patterns are similar to obtained under the usual conditions, but the pment time is markedly reduced. Centri- and electrical forces may be applied inde- ntly, in sequence or simultaneously.
K. A. Proctor

8. Some artefacts in paper chromatography. weig (Univ. of California, Davis, U.S.A.). *Chem.*, 1959, **31** (5), 821–824.—Artefact have been observed on paper chromatograms ino acids, organic acids and bases, and in- c ions. Some are due to esterification by, or ex formation with, the solvent system; others ue to multiple ionic species and geometrical rs. Corrective measures are recommended to ise their occurrence. A. R. Rogers

9. Quantitative evaluation of chromatograms ouble decomposition with simple radioactive its. I. The use of $Ag^{131}I$ in radiometric ation of organic and phosphoric acids. Z. J. ycki, A. Z. Budzyński and I. G. Campbell . of Radiobiol. and Radiochem., Inst. of ar Res., Warsaw, Poland). *Talanta*, 1959, 165–170.—The chromatograms of fatty acids c, myristic, palmitic and stearic), of phos- s (ortho-, pyro-, trimeta- and hypo-phosphates) f glucose 1-phosphate are obtained by specified dures and air-dried. They are then hung in cial vessel made of paraffin wax, and treated mersion with satd. aq. Ag acetate soln. for n. After being washed with water (3 × 5 ml), are treated by immersion for 15 min. with *M* aq. KI containing 3 μC of $K^{131}I$ per ml, washed thrice with water as above, and ed to dry in the dark at room temp. Activity measurements are made in a previously described instrument (Budzyński *et al.*, "Second U.N. Int. Conference on Peaceful Uses of Atomic Energy," P/1588, Geneva, 1958), with a slit width of 2 mm. The integrated activities of the spots are linearly related to spot content, and the maximum (peak) spot activity is proportional to the log. of spot content for fatty acids and phosphates, but not for glucose 1-phosphate. Errors are as follows for the loads stated—fatty acids, 4·6 to 7·1% (50 to 400 μg); phosphates, 4·9 to 11·2% (50 to 500 μg); glucose 1-phosphate, 1·9% (20 to 300 μg).
R. E. Essery

5030. **Carrier displacement chromatography on ion-exchange resins.** D. L. Buchanan (Radio-isotope Service, Veterans Admin. Hosp., West Haven, Conn., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 833–836.—Amino acids may be separated by differential migration into and through a zone of resin which has first been saturated with an appropriate acid or base. The method has been applied to the isolation of free amino acids from picric acid extracts of rat muscle. The capacity of a given column and the concn. of amino acid in the percolate are greater when the carrier is a weak base than when mineral acids or buffers are used.
A. R. Rogers

5031. **Gas chromatography.** N. H. Ray (Plastics Division, I.C.I. Ltd., Welwyn Garden City, England). *J. Soc. Glass Tech.*, 1959, **43**, 100T–112T.—A review of methods and apparatus is given, with 25 references. J. A. Sugden

5032. **Fractional co-distillation in gas-chromatography apparatus.** G. H. Cady and D. P. Siegwarth (Dept. of Chem., Univ. of Washington, Seattle). *Anal. Chem.*, 1959, **31** (4, Part I), 618–620.—A method of separating small amounts of volatile substances is described, in which the separation is dependent upon liquid - vapour (or solid - vapour) equilibria, a temp. gradient in a packed column and a rising temp. in the column as a whole. The separated substances emerge in the order of decreasing volatility and substances may be separated which react with the type of packing usually used in gas chromatography. Substances which sublime may also be separated. K. A. Proctor

5033. **Analysis of aqueous mixtures by gas chromatography.** B. Smith (Inst. f. Organ. Kem., Chalmers Tekniska Högskola, Göteborg, Sweden). *Acta Chem. Scand.*, 1959, **13** (3), 480–488 (in English).—Examples are given illustrating the use of polyethylene glycol 200 and 400, glycerol, diglycerol, hexanetriol, triethanolamine, Tween 80 and glycerol mono-oleate as stationary phases for the analysis of aq. soln. of low-molecular-weight alcohols, aldehydes, ketones and carboxylic acids.
W. T. Carter

5034. **Separations by continuous electrochromatography.** A. Karler (Karler Lab., Berkeley, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 848–851.—A general account of the principles and practice of continuous-flow curtain electrophoresis is given and illustrated by the purification of plague toxin and tetanus toxoid. D. G. Moss

5035. **Starch electrophoresis. I. Starch block electrophoresis.** H. Bloemendal (Dept. of Biochem., Netherlands Cancer Inst., Amsterdam). *J. Chromatography*, 1959, **2** (2), 121–135.—A review is presented of apparatus, technique and applications, with 62 references. W. T. Carter

5036. **Improved technique of agar-gel electrophoresis on microscope slides.** R. J. Wieme (Lab. of the Med. Clinic, Univ. of Ghent, Belgium). *Clin. Chim. Acta*, 1959, **4** (3), 317–321.—The electrophoresis tank has end vessels, with platinum electrodes filled with buffer soln., which communicate through large holes with inner vessels filled with buffered agar gel of the same composition as that used for coating the slides. The centre compartment of the tank is filled with light petroleum as cooling fluid. Samples (1 to 3 μl) are introduced into grooves cut in the gel on the slides, which are placed gel downwards so as to form a bridge between the agar blocks; a current of 25 to 35 mA is passed for 25 min. and, after drying, the slides are stained with Amido black 10B (C.I. Acid Black 1). By this technique a minimum of 9 fractions appears in normal human serum. H. F. W. KIRKPATRICK

5037. **Coloured substances as an experimental tool in the study of electrophoretic techniques.** H. Peeters and P. Vuylsteke (St. Jans Hosp., Bruges, Belgium). *Clin. Chim. Acta*, 1959, **4** (1), 58–61 (in English).—The investigation of conditions during electrophoresis with the help of dyes, to aid the development of techniques, is described. H. F. W. KIRKPATRICK

5038. **Electrophoresis tank suitable for wide application.** R. Frentz (Inst. de Biol., Fac. des Sci., Nancy, France). *Clin. Chim. Acta*, 1959, **4** (3), 340–345.—Details are given of the construction of a tank suitable for all types of electrophoresis. H. F. W. KIRKPATRICK

Optical

5039. **Spectrochemical analysis with the help of spectra resolved in time by means of a rotating disc.** Á. Bardócz and R. W. Tarbeling (Spectroscopic Dept., Cent. Phys. Res. Inst., Hungarian Acad. of Sci., Budapest). *Magyar Kém. Foly.*, 1959, **65** (3), 126–128.—At the beginning of spark discharges a strong background is observed in which the weaker lines tend to disappear, *i.e.*, the curves used in spectroscopy become too flat for evaluation. This initial background can be separated by using a rotating sector (which covers part of the length of the slit) and an electronically controlled spark generator, actuated by a photocell. This photocell receives its light from a source through a mirror fixed to the rotating axis of the sector. The amplifying circuit of the cell also includes a phase-shifting device so that the position of the sector with respect to the slit can be varied. The detection of Mn in aluminium at 2949 A is quoted as an example; this method increases the sensitivity by one order of magnitude. A. G. PETO

5040. **Induced-percussion electrode-packing machine.** A. Strasheim and E. J. Tappere (Nat. Phys. Res. Lab., Pretoria, S. Africa). *Appl. Spectroscopy*, 1958, **13** (1), 12–14.—With the machine described, more material can be packed into an electrode cavity than is possible by hand or with a vibrational-type machine. For some materials the reproducibility of the amount packed is better than $\pm$ 1%. K. A. PROCTOR

5041. **Test-tube adapter for Beckman DU spectrophotometer.** B. Zak and L. A. Williams (Wayne State Univ., Detroit, Mich., U.S.A.). *Chemist Analyst*, 1959, **48** (1), 17–18.—To facilitate the measurement of the colours of two im layers, and render their separation unnece wooden cell-holder (illustrated) is constru fit the instrument. A pin passes transversel the hollow in which the cell is inserted, angles to the light-path. When the pi position, it supports the cell so that the low intercepts the light and can be read. W pin is withdrawn, the cell drops on to a c at the bottom of the hollow, and the upp can then be read. During extraction, cent and reading, the cell is closed with a tightl plastic stopper. R. E. Es

5042. **Analytical possibilities of mic spectroscopy.** L. Grifone (Ist. Chim. Fis. di Padova, Italy). *Ric. Sci.*, 1959, **29** (4), 706 The advantages and disadvantages of the te of micro-wave spectroscopy in analytical ch are discussed. It is suggested that a me apparatus incorporating some of the late tronic devices (backward wave oscillator, sensitive crystals and memory tube) shoul value for qual. and quant. determinations. L. ZA

5043. **Infra-red cell assembly for volatile** G. J. Janz and S. S. Danyluk (Dept. of Rensselaer Polytech. Inst., Troy, N.Y., *Appl. Spectroscopy*, 1959, **13** (2), 48–4 simple and inexpensive cell described can for volatile solids when strictly anhydro ditions and controlled atmospheres are esse K. A. PRO

5044. **Window materials for use in i analyses involving nitrogen dioxide.** A. shuller and I. R. Cohen (Public Health U.S. Dept. of Health, Education and Cincinnati, Ohio). *Anal. Chem.*, 1959, **31** I), 628–629.—The effects of 0·17% of n dioxide on calcium fluoride, barium fluori arsenic trisulphide are reported. Calcium windows appear to be completely satisfacto K. A. PRO

5045. **Levelling device for alkali halide disc preparation.** H. W. Morgan (Oak National Lab., Tenn., U.S.A.). *Appl. scopy*, 1959, **13** (2), 48.—The device describ be used to ensure clear, reproducible discs 3 mm in thickness. The levelling of the can be carried out in about 30 sec. and the is easily made from a 5-in. length of brass rod a diameter $\simeq$0·005 in. smaller than the i diameter of the die. K. A. PRO

5046. **Halocarbon oil as a mulling medi infra-red spectra.** D. S. Crocket and Haendler (Univ. of New Hampshire, D U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 62 —Halocarbon oil (series 11–14) has a vi similar to that of Nujol, and is as easy to wor It produces no interference in the region fro to 1300 cm^{-1} and has been used for both in and organic compounds. K. A. PRO

5047. **Electrostatic sample preparator for red spectroscopy of powders.** H. F. Haz (Koninkl./Shell-Lab., Amsterdam). *Spect Acta*, 1959, (1), 1–5.—Strongly coherent film selected particle size may be obtained by elut of powder samples with an inert gas, follo electrostatic precipitation on to a suitable

or. Details of the apparatus are given, and cal spectrum, covering the 2·5 to 15-μ wave- region, of a powdered quartz sample ted on a rock-salt plate is compared with f a similar sample dispersed in a potassium de pellet. P. T. Beale

8. **Report on first X-ray fluorescence round-test.** A.S.T.M. Task Group on X-ray escence Spectroscopy. *Appl. Spectroscopy*, **13** (1), 3–7.—Four factors affecting the ion of X-ray fluorescence spectroscopic ods were examined and gave the following s in terms of coeff. of variation—re-setting erating conditions of the X-ray tube, ± 0·5%; itioning the X-ray spectrometer at the line ⩾ ± 0·5%; estimating composition, ⩾ ± of the amount present; precision of mixing ers to a nominal composition, ± 7%. The tical nature of the X-ray process was found ree experimentally with theory.
K. A. Proctor

9. **A simple photometer.** B. Večerek and J. ková (Inst. Med. Chem., Charles' Univ., ue). *Chem. Průmysl*, 1959, **9** (3), 137–138.— ls are given for the construction of a simple of apparatus suitable for routine photometric ses in the visible range. Ordinary galvano- s used with titrimetric apparatus are suitable e indication of the light intensity. J. Zýka

0. **Micro-photometer for colorimetric titrations.** olasek, H. Lieb and K. Winsauer (Univ., Austria). *Mikrochim. Acta*, 1959, (3), 402–-The micro-photometer described is suitable e exact detection of end-points when carrying olorimetric or nephelometric titrations in vol. 5 to 2·5 ml. Cylindrical cells enable a built-in etic stirrer to be used. D. F. Phillips

1. **Continuous colorimetric analyser and re- r for oxygen control in inert gases.** R. k and A. Rečka (Dusíkárny n.p., Ostrava, oslovakia). *Chem. Průmysl*, 1959, **9** (2), 71– The apparatus described has been tested for olling the content of O in nitrogen and in ures of nitrogen and oxygen used in the syn- s of NH_3, and in the production of A. The e is 0·002 to 0·03% (v/v), with an accuracy of 5%. The determination is based on the ption of O in an alkaline soln. of Na anthra- one-2-sulphonate, the reduced form of which l whereas the oxidised form is colourless. The ratus differs from similar types in that the nuous replacement of the soln. is effected by s of a simple regulating device. J. Zýka

2. **Colorimetric apparatus for gas testing. II. ratus for visual determination of oxygen.** acławik and S. Waszak (Anal. Dept., Inst. of Chem., Warsaw). *Chem. Anal., Warsaw*, 1959, -2), 343–349.—The visual colorimetric deter- tion of O in gases is based on the reaction of ith an alkaline soln. of Na anthraquinone-2- onate, which is red when reduced by zinc gam and colourless when oxidised by O nt in the sample of gas. The reagent circulates e apparatus (described and illustrated) in a d circuit, reacting with the gas and then being regenerated in a reducing column. The intensity of the colour of the reagent is proportional to the amount of O. The percentage of O in the sample is determined by bringing a second portion of the reagent to the same colour by treatment with a sample of gas containing a known proportion of O. The apparatus permits the determination of O in the range 0 to 0·1% by vol., but can be adapted to measure quantities $<0{\cdot}01\%$. The time required for a determination is $\simeq 30$ min. and the quantity of gas required is 2 litres.

III. Continuous determination of small quantities of chlorine in gases. S. Waszak. *Ibid.*, 1959, **4** (1–2), 351–359.—The apparatus (described and illustrated) is designed for the continuous visual colorimetric determination of small amounts of Cl (up to 0·010%, or up to 0·001% by vol. with an accuracy of ±10%). The method makes use of the reaction of Cl with an aq. soln. of KI, buffered to pH 7, according to the equation $2KI + Cl_2 = I_2 + 2KCl$. The liberated iodine colours the reagent yellow, the variations in intensity being measured by a differential photometer, and recorded on a calibrated automatic recorder. Oxidising substances that produce similar reactions with KI (*e.g.*, NO_2) must be removed from the sample before examination. W. B. Miaskowski

5053. **Automation in analytical chemistry. II. Apparatus for serial determinations of enzyme activity.** B. Večerek, K. Kácl, L. Kolařík, B. Chundela and J. Večerková (I Inst. Med. Chem., Charles' Univ., Prague). *Chem. Listy*, 1959, **53** (3), 279–280.—A new apparatus is described which enables the colorimetric determination of phosphatases to be carried out automatically. Sodium phenolphthalein phosphate is used as the substrate and the phenolphthalein formed during the course of the reaction is determined colorimetrically in an alkaline medium. The basic part of the described apparatus is constructed from a test-tube transporter by means of which the addition of test soln., buffer and substrate, heating in the thermostat, the addition of the detecting reagent and the measurement of the colour are made automatically.
J. Zýka

Thermal

5054. **Burner with revolving grid.** P. Berthollet. *Chim. Anal.*, 1959, **41** (2), 63–64.—The butane burner described and illustrated permits the simultaneous fusion of 33 specimens without temperature variations. It is compact, the flame is small and gas consumption is reasonable. The apparatus was designed for the preparation of fluoride discs in the fluorimetric determination of U. Results obtained are compared with those given by the type of burner normally in use.
S. M. Marsh

5055. **Recording calorimeter.** J. Pinkava and Z. Večeřa (School of Chem. Technol., Prague). *Chem. Průmysl*, 1959, **9** (3), 135–137.—A new type of calorimeter, which permits the automatic registration of temp. changes, is based on a combination of a Wheatstone bridge with the Kohlrausch drum of a polarograph. The current intensity is measured by means of the galvanometer and is registered photographically. The calorimeter is a Dewar vessel with device for breaking the ampoules containing the sample. The sensitivity of the described

apparatus is higher than that of a Beckmann thermometer and can be used for the registration of changes of 0·001°. J. Zýka

5056. **Torsion thermobalance.** [I]. J. Cueilleron and O. Hartmanshenn (Fac. des Sci., 93 rue Pasteur, Lyons, France). *Bull. Soc. Chim. France*, 1959, **26** (1), 164–168.—A thermobalance is described in which a torsion wire, provided with a beam carrying a counterweight and a pan hanging down into an electric furnace, is mounted in a Pyrex-glass enclosure which can be evacuated. Automatic recording is effected by fitting a shutter to the suspension wire so that it half obscures a photo-electric cell mounted outside the vacuum enclosure. The current from the cell is amplified and fed to a galvanometer with two contacts, which switch on a reversible slow motor (1 rev. per hr.) which twists the torsion head until the shutter is again in the equilibrium position. A pulley on the torsion head connects with the recorder pen. By varying the nature of the torsion wire (0·25-mm-diam. steel piano-wire, 0·15-mm Swedish steel, 0·45-mm or 0·25-mm Elinvar) and the diam. of the pulley, loads from 1 to 15 g can be handled and changes of from 1 to 300 mg can be recorded. The standardisation, use, avoidance of hunting, and theory of the balance are discussed.

II. Application to the decomposition under vacuum of some sulphates. J. Cueilleron and O. Hartmanshenn. *Ibid.*, 1959, **26** (1), 168–172.—The decomposition of a number of sulphates was investigated under continuous pumping in the thermobalance described above. Under these conditions the ordinary hydrates of $CuSO_4$, $CaSO_4$, $CoSO_4$, $NiSO_4$, $MgSO_4$, $ZnSO_4$, $CdSO_4$ and $MnSO_4$, and anhyd. $Fe_2(SO_4)_3$, $CdSO_4$, $SnSO_4$ and $Sn(SO_4)_2$, dehydrate without the formation of a basic salt (except with Zn and Cd) and then (except for Ca) decompose at higher temp. to the oxides which are stable at their temp. of formation (except for CdO which decomposes to Cd and O). At high temp. NiO and CoO undergo a partial loss of O to give a ferromagnetic substance; $CaSO_4$ remains stable up to 970°. Some complex sulphates and alums were also studied under continuous pumping and it was found that $Cr_2(SO_4)_3.18H_2O$ loses $9H_2O$ at room temp., then is dehydrated and begins to decompose to oxide at 150°; $(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$ loses $6H_2O$ at 90°, gives the anhyd. salt and then decomposes to $Fe_2(SO_4)_3$ and then to Fe_2O_3, while $Cr_2(SO_4)_3.K_2SO_4.24H_2O$ loses $6H_2O$ at room temp. and the anhyd. salt (stable from 100° to 400°) decomposes to $Cr_2O_3.K_2SO_4$. A method of quant. analysis of mixtures of sulphates is indicated in which Mg, Mn and Ca individually, or Mg and Mn together, in iron, could thus be determined with an accuracy of 1%.

III. Application to the decomposition under vacuum of some chlorides. J. Cueilleron and O. Hartmanshenn. *Ibid.*, 1959, **26** (1), 172–176.—The thermal decomposition of Recoura's green salt $[CrCl_2(H_2O)_4]Cl.2H_2O$ and violet salt $[Cr(H_2O)_6]Cl_3$ and of $SnCl_2.2H_2O$ has been studied by means of the torsion thermobalance. E. J. H. Birch

5057. **Simple method for derivative differential thermal analysis.** E. S. Freeman and D. Edelman (Pyrotechnics Chem. Res. Lab., Picatinny Arsenal, Dover, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (4, Part I), 624–625.—The method described involves using a duplicate sample as the reference material and maintaining a constant temp. differential in the applied temp. between both samples. Conventional differential thermal analysis apparatus is used. theoretical basis of the method is discussed. K. A. Proc

See also Abstract—**5063**, Differential potentio for measuring changes of temp.

Electrical

5058. **Construction of thread-type salt and immersion-type silver - silver chloride elec** J. T. Stock (Dept. of Chem., Univ. of Connec Storrs, U.S.A.). *Chemist Analyst*, 1959, **4** 8–9.—A thread-type salt bridge is construct drawing 6-mm glass tubing down to a bore of When cooled just to softening-point, an as thread, 10 mm × 0·5 mm (which has been s in HCl, washed and thoroughly dried), is in so that 3 mm projects. Keeping the proj thread as cool as possible, heat is re-applie the tube collapses on the thread, when the e the tube is momentarily pinched with a p pliers so that the end is flattened for about and the end of the asbestos thread is nipp flush with the end of the tube. Only enough to well flatten the tube should be used. practice, fair reproducibility can be achi moderate pinching giving a resistance of 30 5000 ohms and a leak rate, under a 10-cm he 0·03 to 0·05 ml per hr. Harder pinching inc the resistance and decreases the leak-rate factor of about 5. The construction of a si silver chloride electrode embodying such a bridge is described and illustrated. R. E. Esse

5059. **Electrochemical separation process fo in polarography.** R. L. Faircloth (Res. G A.E.R.E., Harwell, Berks., England). *Ta* 1959, **2** (2), 135–151.—If pure bismuth is dissolv mercury and the amalgam placed in contact *M* HCl containing Bi^{3+}, the amalgam takes potential controlled by the relative concn. of the amalgam and Bi^{3+} in the aq. phase, and potential is little affected by trace impuriti the bismuth. If the standard potential of electrode reaction $M(Hg) = M^{n+} + nE$, whe is a metallic impurity, is more negative tha actual amalgam potential, the metal M ten pass into the aq. phase, which can then be exa polarographically. The concn. of Bi^{3+} in th phase can be controlled by adding a known an of HgCl to the soln., whereby Hg^+ are reduc the metal, and an equiv. amount of Bi passes the soln. For the determination of impuriti bismuth, mercury (10 ml) is placed in a glass and covered with 2 ml of *M* HCl containing of HgCl. Bismuth in small pieces (1 g) is dro into the tube, the air is displaced by N, an tube is stoppered and gently shaken, with war till the bismuth has dissolved. After fu vigorous shaking for 15 min., a known vol. o aq. phase is examined in a square-wave polarog in the usual way. The accuracy is ±2%. conditions for the 99·5% extraction of the impu from the bismuth are discussed with referen the Nernst equation, and it is shown that Cd Pb, Tl and Zn can be estimated, but not Cu. determinations referred to are of Zn in cadr and of trace impurities in salt soln. The m can be applied to other metals which are s mercury, and it is also suitable for convent d.c. polarography. R. E. Esse

60. **Rotating sample table for multiple polaro-hic analyses.** J. Luck (Minneapolis-Honeywell ılator Co., Minneapolis, Minn., U.S.A.). *ıist Analyst*, 1959, **48** (1), 18.—The apparatus trated) consists of three metal discs (the upper bored with holes to carry the sample tubes), ıted one above the other on a hollow central , which rotates on a bearing near its top. shaft is fitted with radial tubes, to carry gen through rubber tubes to the bubblers ted in the samples. The reference and dropping ury electrodes are mounted on a stand as a rate unit. The table is rotated to bring each ole tube in turn into position, the bubbler being lifted from the sample tube, the electrode nbly inserted, and the polarogram recorded.
R. E. Essery

61. **Determination of low concentrations by method of differential [subtractive] oscillo-hic polarography.** Yu. M. Kargin (Kazan e Univ.). *Zavod. Lab.*, 1959, **25** (3), 273–276.—n. of Cd, Pb, Bi and Zn of $\simeq 10^{-6}$ *M* are rmined by the use of two, not identical, dropping ury electrodes, the dropping times of which synchronised by means of saw-tooth impulses. iring diagram of the apparatus is given.
G. S. Smith

62. **Use of potential compensators in polaro-hic titrations.** L. Klír, K. Liška and Z. Tischer . Res. Ores, Prague). *Chem. Listy*, 1959, **53** 432–433.—A new device has been suggested olarographic titrations for use with a rotating num electrode and a S.C.E. A resistance is ched into the circuit instead of the galvano-er and the resulting potential is measured with otential compensator. The method has been ed on the titration of As^{3+} (0·0001 *N*) with O_3.
J. Zýka

63. **An electronic recording differential potentio-r.** C. Solomons and G. J. Janz (Dept. of Chem., sselaer Polytech. Inst., Troy, N.Y., U.S.A.). *l. Chem.*, 1959, **31** (4, Part I), 623.—The equip-t described has been used to record cooling es during cryoscopic experiments on molten soln. in the temp. range 200° to 400°. Temp-ures may be read off the recorder chart to the est 0·01°. The final 3° of the cooling curve is ad over 4 inches of chart. In measuring the in temp. of a calorimeter when determining heats of fusion of organic salts, temp. changes 1° can be readily measured to within 0·005°.
K. A. Proctor

64. **Electronic controlled-potential coulometric tor. I.** M. T. Kelley, H. C. Jones and D. J. er (Oak Ridge National Lab., Tenn., U.S.A.). *l. Chem.*, 1959, **31** (4, Part I), 488–491.—The tor can be used for either oxidation or reduction ometric titrations with good precision and can sed either automatically or manually.
. M. T. Kelley, H. C. Jones and D. J. Fisher. , 1959, **31** (5), 956.—Circuit changes have made to improve the performance of the ronic controlled-potential coulometric titrator ribed in Part I.
K. A. Proctor

65. **Use of molecular filter membrane in nting and assaying of radioactive precipitates.** . Jervis (Chem. and Met. Div., Atomic Energy of Canada Ltd., Chalk River, Ontario). *Talanta*, 1959, **2** (1), 89–91.—Molecular filter membrane has a much lower weight per unit area than filter-paper and undergoes no detectable loss in weight when heated for 30 min. at 120°. It is more suitable than filter-paper for assaying radioactive ppt., but must be used with care since it has a lower mechanical strength and is soluble in some organic solvents. A machined Teflon filtration apparatus for use with molecular filter membranes is illustrated.
W. T. Carter

5066. **Photomultiplier window for a liquid scintillator.** R. M. Tennent (Univ. of Leeds, England). *J. Sci. Instrum.*, 1959, **36** (3), 148.—The glass of a 2·5-in. cylindrical glass - metal seal is cut 0·75 in. from the seal and a flat glass plate sealed to the end. The metal is soft-soldered directly to the stainless steel of the scintillator tank. The exposed portion of the metal (Kovar) tube and the solder joint are painted with a mixture of titanium oxide and water-glass to avoid the risk of poisoning the scintillator.
G. Skirrow

5067. **Precision calibration of large 4π scintillation crystals for routine γ-counting.** L. J. Colby, jun., and J. W. Cobble (Dept. of Chem., Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 798–799.—A large well-type scintillation crystal counter is calibrated by the 4π β-γ coincidence method and can then be used as a secondary standard for calibrating electron-capture isotopes with a much higher accuracy ($\pm$ 3%) than was previously possible. The system is applicable to the analysis of mixed isotopes and of decay schemes.
K. A. Proctor

5068. **Absolute β standardisation using 4π β-γ coincidence techniques.** R. Gunnink, L. J. Colby, jun., and J. W. Cobble (Dept. of Chem., Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 796–798.—The use of the 4π β-γ coincidence counter is recommended as a primary β-standardising method as it is free from many disadvantages but requires a true β-γ coincidence. The 4π method can be evaluated against this procedure.
K. A. Proctor

5069. **Evaluation of the Cenco β-ray H/C meter. A new calibration procedure.** R. Rowan, jun. (Products Res. Div., Esso Res. and Engng Co., Linden, N.J., U.S.A.). *Anal. Chem.*, 1958, **31** (4, Part I), 558–561.—A new method of determining the fundamental instrument constant, K_H, is described. The precision lies in the range of 0·01 to 0·02% of H for pure or mixed hydrocarbon samples. The accuracy depends on the exactness of the calibration.
K. A. Proctor

5070. **Direct estimation of γ-ray abundances in radionuclide mixtures. Complement subtraction method.** W. Lee (U.S. Naval Radiolog. Defense Lab., San Francisco, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (5), 800–806.—γ-Emitting radionuclides can be determined in mixtures by a direct rapid technique which combines the use of an Argonne-type 256-channel pulse-height analyser with a dead-time correcting device. Pure calibrated standards are required for each radionuclide and this limits the accuracy of the method for absolute quant. work.
K. A. Proctor

5071. **Sensitivity charts for neutron activation analysis.** W. W. Meinke (Univ. of Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1959, **31** (5), 792–795.—Sensitivity charts are given for a number of elements irradiated by thermal neutrons for periods of 6 min., 10 hr. and 1000 hr., and are useful for comparing the sensitivities of this and other methods of analysis. K. A. PROCTOR

5072. **Chemical analysis by neutron activation.** J. Fouarge (Lab. Chim. Anal., Univ. Liége, Belgium). *Ind. Chim. Belge*, 1959, **24** (2), 143–154.—The theory and limitations of the method, which is sensitive enough for the detection of most elements in the 10^{-5} to 10^{-15}-g range, are discussed, and applications are briefly reviewed. A table of sensitivities is included. (75 references.)

[S.C.I. ABSTR.

5073. **An alarm for the detection of tox dustrial gases.** A. M. Kinnear (Chem. D Expt. Estab., Porton Down, Salisbury, V England). *Chem. & Ind.*, 1959, (11), 361–3 The air is drawn through an illuminated a filter-paper tape and the production of a col reaction product reduces the amount of ref light received by a photocell and so trips the a A built-in reagent pump can be used to impre the paper with up to three separate reagen alternatively, a dry, untreated or prev impregnated tape may be used. Approx. lim detection are 0·3 p.p.m. of H_2S, 0·5 p.p.m. of 0·9 p.p.m. of $COCl_2$, 0·1 p.p.m. of Cl and 1·5 p of SO_2. K. A. PROC

See also Abstracts—**4983**, Portable analys dissolved O in water. **4991**, Automatic titrat sewage.

ERRATA

July (1958) issue, abstract 2272, line 10. *For* "Kircha" *read* "Kirchner."

January (1959) issue, abstract 283, line 2. *For* "Rathbon" *read* "Rathbun."

March (1959) issue, abstract 1006, line 4. *For* "Kirsh" *read* "Kirsch."

April (1959) issue, abstract 1175, last line in second column. *For* "Rease" *read* "Reese."

April (1959) issue, abstract 1285, lines 1 and 5. *For* "chronometric" *read* "chromometric."

July (1959) issue, abstract 2621, line 5. *For* "Bi" *read* "Pd."

July (1959) issue, abstract 2662, line 12. *For* "metal" *read* "metol."

August (1959) issue, abstract 2847, last line. "$KCrO_4$" *read* "K_2CrO_4."

August (1959) issue, abstract 2857, line 5. "2008" *read* "2808."

August (1959) issue, abstract 2943, line 13. "1-(2-pyridyl)azo.1-2-naphthol" *read* pyridyl)azo-2-naphthol."

August (1959) issue, abstract 3073, line 4. "1958" *read* "1959."

September (1959) issue, abstract 3374, line 7. "$Na_3H_2IO_6$" *read* "$Na_2H_3IO_6$."

October (1959) issue, abstract 4196, line 1. "dimeton-methyl" *read* "demeton-methyl."

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any oubt might arise from the use of an abbreviation or symbol the word is printed in full.

lternating current	a.c.
mpere	amp.
ngström unit	A
nhydrous	anhyd.
pproximate, -ly	approx.
queous	aq.
tmospher-e, -ic	atm.
oiling-point	b.p.
British thermal unit	B.Th.U.
alorie (large)	kg-cal.
alorie (small)	g-cal.
entimetre	cm
oefficient	coeff.
oncentrated	conc.
oncentration	concn.
onstant	const.
orrected	(corr.)
rystalline	cryst.
rystallised	cryst.
ubic	cu.
urrent density	c.d.
ycles per second	c/s
lensity	ρ
lensity, relative	d or wt. per ml
lilute	dil.
lirect current	d.c.
listilled	dist.
thylenediaminetetra-acetic acid	EDTA
lectromotive force	e.m.f.
quivalent	equiv.
ram	g
ram-molecule	mole
alf-wave potential	$E_{\frac{1}{2}}$
our	hr.
ydrogen ion exponent	pH
nfra-red	i.r.
nsoluble	insol.
nternational unit	i.u.
ilogram	kg
ilovolt	kV
ilowatt	kW
iquid	liq.
naxim-um, -a	max.
nelting-point	m.p.
nicrogram	μg (not γ)
nicrolitre	μl
nicromole	μmole
nicron	μ
nilliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or n mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
s proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the dentifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

Volume 6. No. 12, Abstracts 4655–5073 December, 19

ANALYTICAL ABSTRACTS

A PUBLICATION OF
THE SOCIETY FOR ANALYTICAL CHEMISTRY

EDITORIAL COMMITTEE

Chairman: B. A. Ellis

Members: B. S. Cooper, C. H. R. Gentry, C. A. Johnson, H. F. W. Kirkpatrick, B. J. Walby, W. A. Waygood, K. A. Willian
and
President of the Society: R C. Chirnside

Hon. Secretary of the Society: R. E. Stuckey

Hon. Treasurer of the Societ A. J. Amos

Editor: Norman Evers, B.Sc., Ph.D., F.R.I.C.

Assistant Editor: Mrs. H. I. Fisk, B.S

CONTENTS

Abstr

General Analytical Chemistry

Reviews; reagents; methods of general application 4

Inorganic Analysis

General methods; elements (in order of the Periodic Table); minerals; industrial products 4

Organic Analysis

Elements and radicals; organic compounds; industrial products 4

Biochemistry

BIOLOGICAL FLUIDS; ANIMAL AND VEGETABLE TISSUES 4

PHARMACEUTICAL ANALYSIS 4

FOOD

Food additives; beverages; edible oils and fats; vitamins 4

SANITATION

Air; water; sewage; industrial wastes 4

AGRICULTURAL ANALYSIS

Soil; fertilisers; herbicides; pesticides; animal feeding-stuffs 4

General Technique and Apparatus

GENERAL 5

CHROMATOGRAPHY; ION EXCHANGE; ELECTROPHORESIS 5

OPTICAL 5

THERMAL 5

ELECTRICAL 5

Printed and published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, Engl
Communications to be addressed to the Editor, Norman Evers, 14, Belgrave Square, London, S.W.1
Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.
Entered as Second Class at New York, U.S.A., Post Office.